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NOTCH SENSITIVITY OF REFRACTORY METALS

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(Prepared under Contract No. AF 33(616)-7604
by Battelle Memorial Institute, Columbus, Ohio;
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FOREWORD

This report was prepared by Battelle Memorial Institute under USAF Contract No. AF 33(616)-7604. This contract was initiated under Projects 1(8-7351) and 1(8-7381), and Tasks Nos. 73521 and 73812. The project was administered under the direction of Directorate of Materials and Processes, Deputy for Technology, Aeronautical Systems Division, with Lt. Ben A. Wilcox and Lt. Robert Ault acting as project engineers.

This report covers the period from October 1, 1960, to August 31, 1961.

ABSTRACT

The effects of interstitial oxygen and hydrogen on the tensile and notch tensile properties of tantalum and columbium were investigated. The tensile and notch tensile properties of Ta-10W and F-48 columbium alloy were determined also. Oxygen and hydrogen additions resulted in notch-sensitive behavior at higher temperatures than for pure Ta and Cb, and, similarly, transition temperatures are increased by the interstitial additions. The F-48 alloy also shows notch-sensitive behavior at higher temperatures and has a higher transition temperature than columbium. The Ta-10W alloy was not notch sensitive, and retained excellent ductility at -420 F.

In addition, reaction kinetics in the tantalum-hydrogen system were studied.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

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NOTCH SENSITIVITY OF REFRACTORY METALS

by

A. G. Imgram, M. W. Mallett, B. G. Koehl,
E. S. Bartlett, and H. R. Ogden

INTRODUCTION

Strength or oxidation-protection problems generally restrict the use of refractory metals at elevated temperatures. However, at low temperatures lack of ductility and notch sensitivity are of primary concern. This is of importance where structural parts are used at low temperatures and/or are subjected to multiaxial stresses.

The tensile transition from ductile to brittle behavior and notch sensitivity of wrought and recrystallized, high-purity (electron-beam melted) tantalum and columbium were investigated by Battelle Memorial Institute under Contract No. AF 33(616)-6291, entitled "Notch Sensitivity of Refractory Metals".[1] No serious problems of brittle behavior or notch sensitivity were detected in wrought or recrystallized tantalum above -420 F or with columbium above -323 F. However, it was thought that higher interstitial contents, such as might be expected in some commercial-purity grades of these materials, might promote brittle behavior and notch sensitivity at higher temperatures than in the high-purity bases. The embrittling effects of interstitial-impurities atoms are usually related either to (1) their interaction with dislocations, greatly increasing the yield strength at low temperatures, (2) their segregation in grain boundaries forming a brittle grain-boundary film, or (3) the formation of a hard, brittle second phase.

This research program was conducted to determine the effects of interstitial oxygen and hydrogen on the transition behavior and notch sensitivity of wrought and recrystallized tantalum and columbium. All interstitial additions were kept within solid-solubility limits. Therefore, it was expected that, observed embrittling effects would largely be due either to interactions with dislocations, or grain-boundary segregation.

In addition, substitutional alloying elements may not only increase the strengths of tantalum and columbium, but may also contribute to low-temperature brittleness. This research program also evaluated the notch sensitivity of one tantalum alloy (Ta-10W) and one columbium alloy (F-48).

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(1) References are given on page 75.

Related to this effort was a study of thermal equilibria and reaction kinetics in the tantalum-hydrogen system. One section of this report is devoted to a description of the findings of this study.

SUMMARY

The effects of interstitial oxygen and hydrogen on the tensile and notch tensile properties of tantalum and columbium were investigated. Commercially pure Ta-10W and F-48 columbium alloy were also studied. Each material and condition was evaluated both in wrought and recrystallized structural conditions. Oxygen and hydrogen additions were made in a Sieverts apparatus and were kept within solid-solubility limits. Oxygen levels considered were 500 and 1000 ppm in both tantalum and columbium. Hydrogen contents of 150 and 500 ppm were selected for tantalum whereas levels of 150 and 300 ppm were selected for columbium. Notched and unnotched tensile specimens were tested over a range of temperatures selected to encompass the ductile-to-brittle transition. The notch sensitivity and transition behavior of each material and condition were evaluated largely on the basis of notch-unnotch strength ratio and ductility transition. The effects of oxygen and hydrogen on the tensile properties of tantalum and columbium were evaluated by comparing the data of oxygenated and hydrogenated material with data determined for the pure tantalum and columbium under the previous contract of this research program.[1]

Tantalum

Increasing the oxygen content of tantalum increased its strength and raised its ductility transition temperature, but did not generally cause a serious deterioration of notch properties at temperatures as low as -420 F. Oxygen contents above 500 ppm severely reduced ductility. The adverse effects of oxygen were more pronounced for recrystallized structures than for wrought material.

Increasing the hydrogen content of tantalum increased its strength slightly. The effects of hydrogen content on the ductility transition temperature and notch sensitivity were confused by what was thought to be a strain-aging phenomenon causing low ductility and poor notch properties at -105 F. Nevertheless, hydrogen severely reduced the ductility of tantalum. The effects of hydrogen were comparable on wrought and recrystallized structures.

Columbium

Increasing the oxygen content of columbium increased its strength and raised its ductility transition temperature. Evidence of notch sensitivity was observed at increasingly higher temperatures as the oxygen content was raised. Similar to what was observed for tantalum, adverse effects of oxygen were more pronounced for recrystallized structures than for wrought material.

Increasing the hydrogen content of columbium had only a moderate effect on strength. The ductility transition temperature was raised above room temperature when the hydrogen content was increased to 150 ppm. However, a further increase in hydrogen content did not appear to significantly affect the transition behavior. Similar to what was observed for tantalum, evidence of a strain-aging phenomenon resulting in poor notch properties was detected at -105 F.

Ta-10W Tantalum Alloy

No serious problems of brittle behavior or notch sensitivity were detected at temperatures as low as -420 F.

F-48 Columbium Alloy

Recrystallized F-48 exhibited evidence of notch sensitivity and brittle behavior slightly above room temperature. Wrought F-48 became brittle and notch sensitive slightly below room temperature.

Reactions in the Tantalum-Hydrogen System

Equilibria and thermodynamic functions were determined for the atom fraction (N_H) 0.05, 0.10, 0.15, 0.20, 0.25, 0.30, and 0.333 of the tantalum-hydrogen system. The solid consisted of a single-phase solution for the experimental temperature range, 300 to 700 C (570 to 1290 F). Kinetics of reaction of hydrogen with tantalum was studied for compositions $N_H = 0.05$ and $N_H = 0.10$, at 300 to 700 C. The process was one of dissolution and diffusion of hydrogen. The diffusion coefficients obtained are expressed by the following equations:

$$D(N_H = 0.05) = 305 \exp[(-29{,}460 \pm 1540)/RT] \ cm^2/sec$$

$$D(N_H = 0.10) = 3790 \exp[(-31{,}460 \pm 1030)/RT] \ cm^2/sec$$

Scattered rates at $N_H = 0.25$ and $N_H = 0.333$ also were determined.

PART I. INVESTIGATION OF TENSILE AND NOTCH TENSILE PROPERTIES

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EXPERIMENTAL PROCEDURES

Materials

The high-purity tantalum and columbium used as base material in this research program were purchased as 1/2-inch-diameter bar stock from the Wah Chang Corporation. Ta-10W and F-48 columbium alloy were purchased as 1/4-inch-diameter rod from the National Research Corporation and the General Electric Company, respectively. The manufacturers' fabrication procedures are described in Table 1. Available analytical data are reported in Table 2.

Oxygenation and Hydrogenation

Oxygen levels selected for study were 500 and 1000 ppm in both tantalum and columbium. Hydrogen additions of 150 and 500 ppm in tantalum, and 150 and 300 ppm in columbium were chosen. The higher interstitial contents were selected below the expected room-temperature solid-solubility limits. The lower value provided a comparison of transition behavior with an intermediate interstitial content between the solid-solubility limit and the high-purity base material. No oxygen or hydrogen additions were made to the two alloys, Ta-10W and F-48.

All oxygen and hydrogen additions were made directly to 6-inch lengths of the 1/2-inch-diameter bar stock in a Sieverts apparatus. This equipment is basically an electric resistance-wound furnace in which an accurately measured volume of gas can be reacted with the charge material. The residual gas pressure is negligible. Oxygen additions were made in this manner.

In the case of hydrogen, a hydrogen atmosphere was maintained at precisely the equilibrium pressure for the desired content at the reacting temperatures.

TABLE 1. FABRICATION HISTORY OF TANTALUM AND COLUMBIUM

Tantalum	Columbium
(1) Electron-beam melt ingot 5 inches in diameter	(1) Electron-beam melt ingot 5 inches in diameter; are melt to 9 inches in diameter
(2) Cold forge to 1-5/8-inch-diameter round bar	(2) Forge at 2300 F to 1-5/8-inch-diameter round rod.
(3) Scalp 1/4 inch from all surfaces	(3) Red air cooled to room temperature
(4) Red cold swage to size with reductions of 10 per cent per pass	(4) Scalp 1/4 inch from all surfaces
(5) Final condition by pickling	(5) Swage at 1450 F with 10 per cent reduction per pass
	(6) Final condition by pickling

Ta-10W	F-48
(1) Slow arc melt (0.5-1 pound/min) for purification	(1) Arc melt ingot
(2) Fast arc melt (3-5 pound/min) for homogenization and grain refinement	(2) Extrude at 2800 F to 1-1/4 inch by 4 inch sheet bar
(3) Forge to 1.5 inch diameter rod at 2100 F	(3) Machine 1-inch-diameter slugs
(4) Vacuum anneal 1 hr at 3000 F	(4) Anneal for 1 hr at 2800 F
(5) Cold roll to 0.250-inch-diameter rod without intermediate anneals	(5) Cool with flame-sprayed molybdenum for lubrication
	(6) Impact extrude at 2700 F to 9/16-inch-diameter rod
	(7) Centerless grind to 0.375 inch diameter to remove scale and imperfections
	(8) Swage to 0.290 inch diameter at 2100 F
	(9) Centerless grind to 1/4 inch diameter
	(10) Final condition by pickling

TABLE 2. CHEMICAL ANALYSIS OF BASE MATERIALS AND ALLOYS

Parts per Million

	Tantalum	Columbium	Ta-10W	F-48
Al	< 20	< 20	--	--
B	< 1	< 1	--	--
C	< 30	50	15	200-400
Cb	< 500	Major	--	--
Cd	< 5	< 5	--	--
Cr	< 20	< 20	10	--
Cu	< 40	< 40	--	--
Fe	< 100	< 100	50	--
H_2	< 2	< 2	--	--
Hf	--	< 80	--	--
Mg	< 20	< 20	--	--
Mn	< 20	< 20	--	--
Mo	< 20	< 20	25	5.0-5.2%
N_2	14	48	25	200-350
Ni	< 20	< 20	50	--
O_2	< 50	60	110	200-500
Pb	< 20	< 20	--	--
Si	< 100	< 100	25	--
Sn	< 20	< 20	--	--
Ta	Major	< 500	--	--
Ti	< 150	< 150	10	--
V	< 20	< 20	--	--
W	< 300	< 300	--	15-16%
Zn	< 20	< 20	--	--
Zr	< 500	< 500	--	1.0-1.4%

Six 6-inch lengths of each material were prepared at each selected interstitial level. Before placing in the Sieverts apparatus, the bars were surface abraded and degreased with acetone. The Sieverts apparatus treatment is described in Table 3. A section was cut from an end of one bar of each material after treatment in the Sieverts apparatus. Hardness traverses were made across one section diameter of each test slug. These measurements revealed that the oxygen in the oxygenated tantalum and columbium bars was essentially confined to the surface regions. Therefore, these bars were homogenization annealed in vacuum. A hardness traverse across a section diameter of the annealed bars indicated that the oxygen was uniformly distributed. The hardness measurements of the hydrogenated bars indicated that the hydrogen was uniformly distributed after treatment in the Sieverts apparatus, and no homogenization anneal was required.

Microstructural examination at room temperature did not reveal the presence of a second phase in any of the oxygenated or hydrogenated bars.

Fabrication

After oxygen and hydrogen additions were made, the 1/2-inch-diameter bars were swaged to 1/4-inch-diameter rods. Fabrication procedure for each material and interstitial content is described in Table 4.

Both tantalum and columbium with the lower levels of oxygen (500 ppm) and hydrogen(150 ppm) were swaged at room temperature. However, to obtain sound material with the higher interstitial contents, it was necessary to swage at elevated temperatures. The tantalum and columbium bars with 1000 ppm O_2 added, were sealed in stainless steel "cans" under an argon atmosphere and swaged at 1800 F. Tantalum and columbium with 500 and 300 ppm H_2, respectively, were swaged at 500 F. It was not necessary to protect the materials at this temperature.

The Ta-10w and F-48 columbium alloy rods were purchased as 1/4-inch-diameter rod, and therefore did not require fabrication.

Heat Treatment

Each material and interstitial level was evaluated in the wrought stress-relieved, and recrystallized structural conditions. The stress-relief heat treatment was selected just below the start of recrystallization to maintain a completely wrought structure with maximum ductility. The recrystallization anneal was chosen to provide a fully recrystallized structure with

TABLE 3. OXYGENATION AND HYDROGENATION OF TANTALUM AND COLUMBIUM

Interstitial Level	Treatment in Sieverts Apparatus	Homogenization Anneal	Average Hardness, VHN
	Tantalum		
500 ppm O_2	Reacted with oxygen gas for 17 hr at 1650 F	3 hr at 3275 F	243
1000 ppm O_2	Reacted with oxygen gas for 22 hr at 1650 F	3 hr at 3275 F	162
150 ppm H_2	Equilibrium pressure maintained for 150 ppm H_2 level for 15 hr at 930 F	--	212
500 ppm H_2	Equilibrium pressure maintained for 500 ppm H_2 level at temperature from 1300 to 500 F	--	--
	Columbium		
500 ppm O_2	Reacted with oxygen gas for 5-1/2 hr at 1650	16 hr at 2370 F	163
1000 ppm O_2	Reacted with oxygen gas for 9 hr at 1650 F	16 hr at 2370 F	142
150 ppm H_2	Equilibrium pressure maintained for 150 ppm H_2 level for 18 hr at 930 F	--	161
300 ppm H_2	Equilibrium pressure maintained for 300 ppm H_2 level at temperatures from 1300 to 750 F	--	--

TABLE 4. FABRICATION OF OXYGENATED AND HYDROGENATED TANTALUM AND COLUMBIUM

Interstitial Level	Swaging Temperature, F	Hardness, VHN	Interstitial Analyzer, ppm O_2	Interstitial Analyzer, ppm H_2
		Tantalum		
500 ppm O_2	Room temperature	227	524	<1
1000 ppm O_2	1800 F	257	947	<1
150 ppm H_2	Room temperature	198	12	137
500 ppm H_2	500 F	187	68	587
		Columbium		
500 ppm O_2	Room temperature	161	600	<1
1000 ppm O_2	1800 F	163	1320	3
150 ppm H_2	Room temperature	148	66	105
300 ppm H_2	500 F	151	201	450

a minimum of grain growth. Specimen blanks were cut from the 1/4-inch-diameter swaged rods and were heat treated as outlined in Table 5. Ta-10W, F-48, and oxygenated tantalum and columbium were annealed in a vacuum tube furnace. However, most of the hydrogen in the hydrogenated tantalum and columbium would have diffused out of the specimen blanks if they were heat treated in vacuum. Therefore, they were heat treated under a hydrogen atmosphere in a Sieverts apparatus where hydrogen equilibrium pressures for the desired interstitial hydrogen content were maintained during heating and cooling. Equilibrium pressures were maintained by adding or extracting hydrogen gas from the system as necessary. Unfortunately, the apparatus broke as the recrystallized tantalum specimens with 500 ppm hydrogen were being cooled from the annealing temperature, and some hydrogen was lost. The analyzed hydrogen content (271 ppm) was less than the desired value.

Grain sizes of the recrystallized oxygenated and hydrogenated tantalum and columbium compared closely to those of the pure material studied under the previous contract(1). Therefore the data could be analyzed to determine the effects of interstitial oxygen and hydrogen with a minimum of complications due to variations in grain size. The grain structure of the columbium specimens was quite uniform. However, the tantalum specimens exhibited rather large differences in grain size. There were areas mainly near the central axes of the rods where there were several large grains surrounded by many small grains. This condition is probably the result of nonuniform deformation. It was impossible to develop a more uniform structure by heat treatment. Increasing the recrystallization temperature appeared to aggravate the condition.

Microstructural examination did not reveal the presence of a second phase. Therefore it was assumed that all oxygen and hydrogen additions were within the solid-solubility limits as planned. Photomicrographs of each test condition are presented in later sections of this report dealing with mechanical properties.

Machining

Heat-treated specimen blanks were machined to the specifications described in Figure 1. Notched specimens were designed with a 60-degree notch angle, a 50 per cent notch depth, and a 0.006-inch notch root radius resulting in a theoretical stress concentration (K_t) of approximately 3 at the base of the notch. The specimen diameter at the base of the notch was 0.125 inch. Unnotched specimens were prepared with the same reduced section as that at the root of the notched specimens and had a 1/2-inch gage length.

Accuracy and smoothness of the notch root radius was insured by a hand-lapping operation. A 0.012-inch-diameter tungsten wire, coated with

TABLE 5. HEAT-TREATMENT SCHEDULE

Nominal Interstitial Level	Condition	Heat Treatment	Hardness, VHN	Interstitial Analyses, ppm O_2	Interstitial Analyses, ppm H_2	Grain Size, ASTM
		Tantalum				
500 ppm O_2	Stress relieved	1 hr at 1380 F	219			
	Recrystallized	3 hr at 2190 F	194	489	5	2-4
1000 ppm O_2	Stress relieved	1 hr at 1380 F	255			
	Recrystallized	1 hr at 2550 F	247	758	4	2-4
150 ppm H_2	Stress relieved	1 hr at 1380 F	154	77	145	
	Recrystallized	3 hr at 2190 F	86	86	125	2-4
500 ppm H_2	Stress relieved	1 hr at 1380 F	153	190	582	
	Recrystallized	3 hr at 2190 F	91	23	271	2-4
		Columbium				
500 ppm O_2	Stress relieved	1 hr at 1380 F	151			
	Recrystallized	1 hr at 2190 F	114	484	11	5-6
1000 ppm O_2	Stress relieved	1 hr at 1380 F	161			
	Recrystallized	1 hr at 2190 F	152	1320	21	6-7
150 ppm H_2	Stress relieved	1 hr at 1380 F	121	27	191	
	Recrystallized	1 hr at 2190 F	77	218	213	3-5
300 ppm H_2	Stress relieved	1 hr at 1380 F	123	183	384	
	Recrystallized	1 hr at 2190 F	88	209	395	8
		Ta-10W				
Pure	Stress relieved	1 hr at 2190 F	255			
	Recrystallized	1 hr at 2730 F	216	49	3	6-7
		F-48				
Pure	Stress relieved	1 hr at 2200 F	267			
	Recrystallized	1 hr at 2912 F	270			

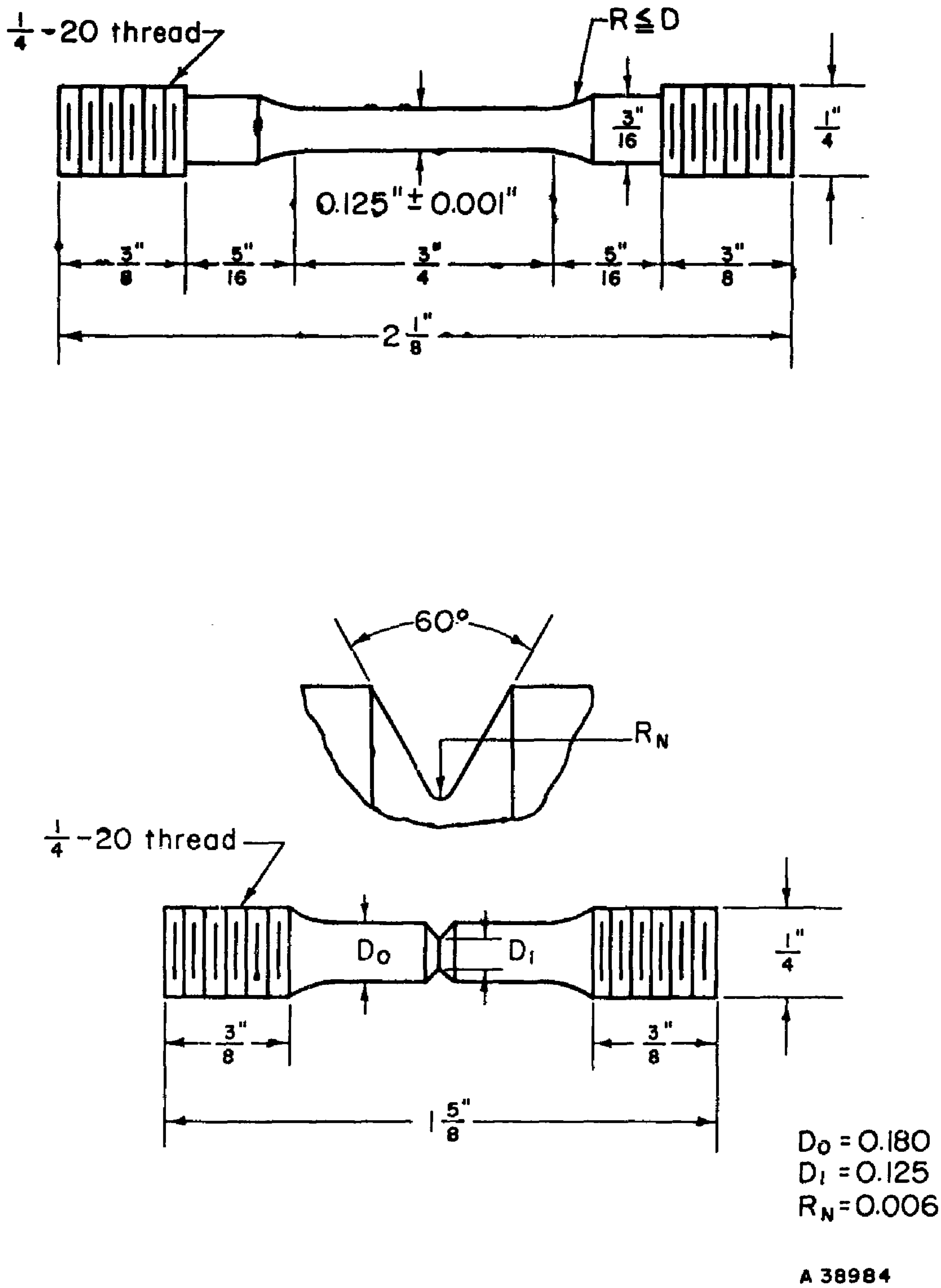


FIGURE 1. NOTCHED- AND UNNOTCHED-TENSILE-SPECIMEN SPECIFICATIONS

a mixture of abrasive and machine oil, was held firmly in the base of the notch as the specimen was revolved in a lathe. Dimensions of the notch were then checked on a 50X projection.

Tensile Testing

Notched and unnotched specimens of each material, interstitial level, and structural condition were tested at a series of temperatures selected to encompass the ductile-to-brittle transition.

Testing temperatures of -420 and -323 F were obtained by immersing the specimens in liquid hydrogen and liquid nitrogen, respectively. The boiling points of these liquified gases were taken as the temperature of the tests conducted in these media. An alcohol-dry ice mixture was used to obtain the test temperature of -105 F. This is the lowest temperature obtainable with this mixture. A toluene-filled thermometer was used to measure the temperature.

Elevated-temperature tensile tests were conducted in a resistance-wound, vertical tube furnace. A Chromel-Alumel thermocouple, with the hot junction in contact with the test specimen, connected to a Foxboro controller was used to measure and control the test temperature to within ±5 F.

All tensile tests were conducted in a Baldwin-Southwark Universal Testing Machine. Unnotched specimens were tested at a constant crosshead speed of 0.02 inch per minute, whereas notched specimens were tested at a constant crosshead speed of 0.005 inch per minute. Load-extension curves to the breaking point were obtained for each specimen at all test temperatures. A compressometer (differential transformer), actuated by crosshead movement, fed an electrical impulse into an autographic recorder to plot the elongation component of the curve. The autographic recorder was mechanically geared to the tensile machine's load dial to obtain the load component of the curve. In addition, at room temperature, elongation was determined by measuring the extension of the 1/2-inch gage marks with dividers.

Ultimate tensile strength, 0.2 per cent offset yield strength, reduction in area, and per cent elongation were recorded for each unnotched specimen. For notched specimens, ultimate tensile strength and reduction in area were determined. Point of failure (at or below maximum load) and mechanism of fracture (shear or cleavage) were noted for both notched and unnotched specimens.

The stress-strain relationship for many metals in the plastic region can be described by the equation

$$\sigma = K\delta^n$$

where σ is the true stress, δ is the true strain, n is a constant called the work-hardening exponent, and K is a coefficient equal to the true stress at unit true strain. A plot of true stress versus true strain on logarithmic coordinates (flow curve) then becomes a straight line with slope n. Values of K and n for each material, interstitial level, and structural condition at room temperature were calculated.

Definitions

The nature of the ductility transition does not readily permit the selection of a single temperature above which a material is ductile and below which it is brittle. However, in order to facilitate a simple classification of the effects of oxygen and hydrogen on the transition behavior of tantalum and columbium, definition of the ductility transition has been made as follows:

> The ductility transition temperature is the approximate temperature at which the reduction in area of the material decreases to one-half its maximum valve.

It should be remembered that for some region below this temperature, material usually exhibits considerable ductility.

A similar problem is encountered when attempting to pick a temperature below which a material is notch sensitive. Empirical relationships have shown that the notch-unnotch strength ratio of ductile materials should be approximately 1 + per cent notch depth. Therefore, the 50 per cent notch, as used in this research, should develop a notch strength of 1.5 times that of the unnotch strength when the material is ductile. At temperatures at which the material becomes brittle, the notch-unnotch strength ratio should be less than 1.5. However, this research did not show a sharp demarcation between temperatures at which the materials were not adversely effected by the presence of a notch and those at which the materials were notch sensitive. Again, for use as a guide, the following definition is offered.

> The "notch sensitivity temperature" is the temperature below which the notch-unnotch strength ratio exhibits a relatively rapid decrease with decreasing temperature

When design requirements are demanding, the actual transition and notch-unnotch strength ratio curves should be consulted.

EXPERIMENTAL RESULTS

The figures presented here summarize the notched and unnotched tensile behavior of oxygenated and hydrogenated tantalum and columbium in both wrought (stress relieved) and recrystallized conditions. Typical engineering stress-strain curves are presented in Appendix I. Complete tabulated data are presented in Appendix II.

Tantalum

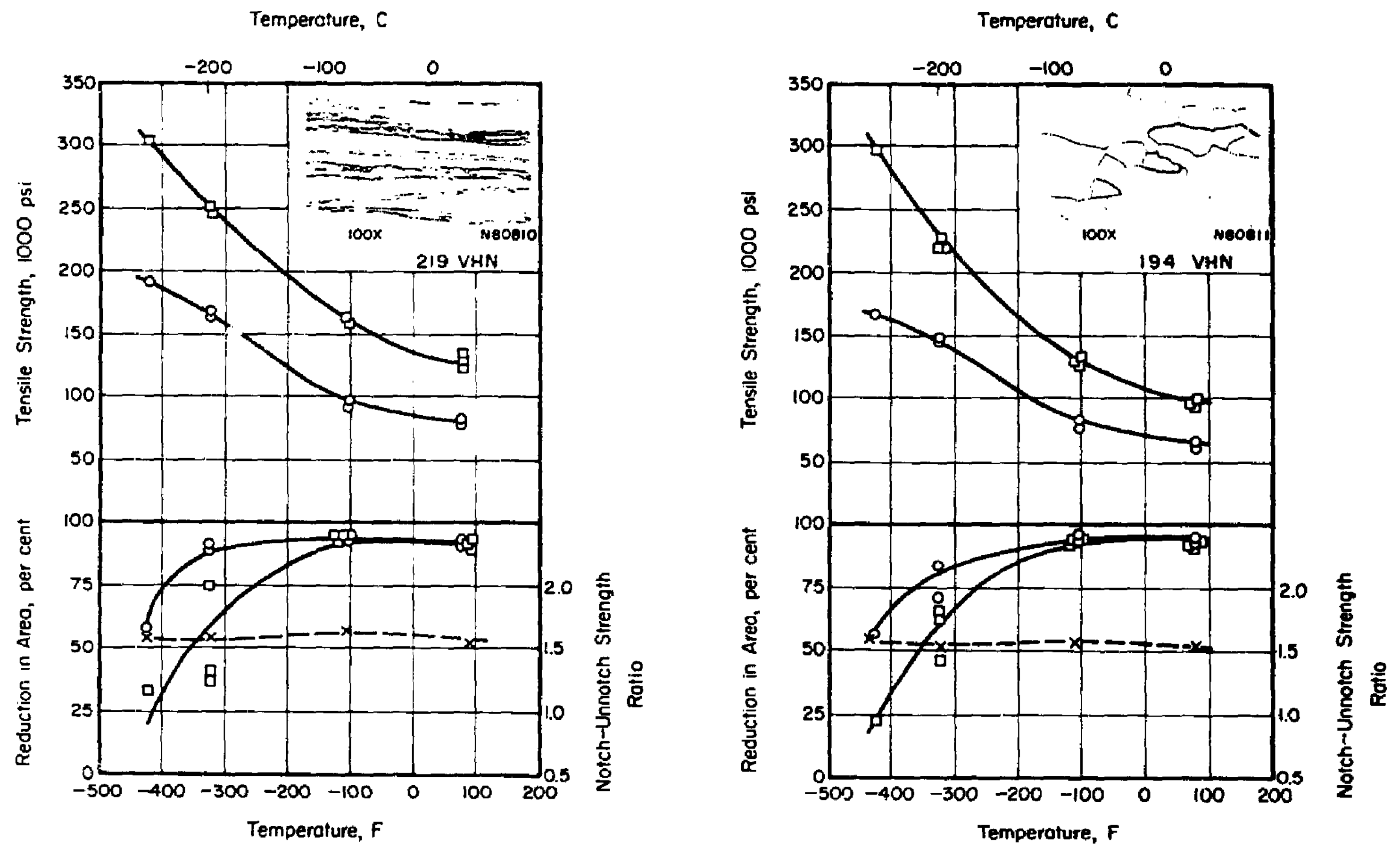
Previous work[1] showed no evidence of brittle behavior or tendency towards notch sensitivity in pure tantalum at temperatures as low as -420 F. Reductions in area remained above 80 per cent at -420 F.

Figures 2 and 3 illustrate the notched and unnotched tensile properties of oxygenated tantalum between 75 and -420 F. With 489 ppm oxygen added, appreciable ductility was retained by both notched and unnotched specimens at -420 F, but ductile-to-brittle transitions were indicated at low temperatures. No evidence of notch sensitivity was detected. With the higher oxygen content (758 ppm) ductility transition temperatures were appreciably higher for the recrystallized specimens than for the wrought material. Surprisingly, the recrystallized material did not show evidence of notch sensitivity whereas the wrought material did.

The tensile properties of tantalum containing two levels of hydrogen are illustrated in Figures 4 and 5. The most striking feature is that at -105 F minimum points were generally observed in notched and unnotched ductilities. This was interpreted to be a strain-aging phenomenon, and a principal cause of inferior notch behavior at -105 F. Normal transition behavior was obscured by this effect. At the low hydrogen level, notch properties and ductility recovered, to some extent, at lower temperatures. Recovery was less pronounced at the high hydrogen level. It should be remembered that at the "high" hydrogen level (Figure 5) the recrystallized specimens contained considerably less hydrogen than the wrought material as explained previously.

Columbium

Previous research[1] showed that both wrought and recrystallized columbium had ductile-to-brittle transitions at approximately -400 F. Notched specimens in both conditions exhibited no ductility at -420 F.



a. Wrought

b. Recrystallized

A-38985

Legend

- o Unnotched specimens
- □ Notched specimens (K_t=3)
- x Notch-unnotch strength ratio

FIGURE 2. TENSILE AND NOTCH TENSILE PROPERTIES OF TANTALUM WITH 489 PPM OXYGEN

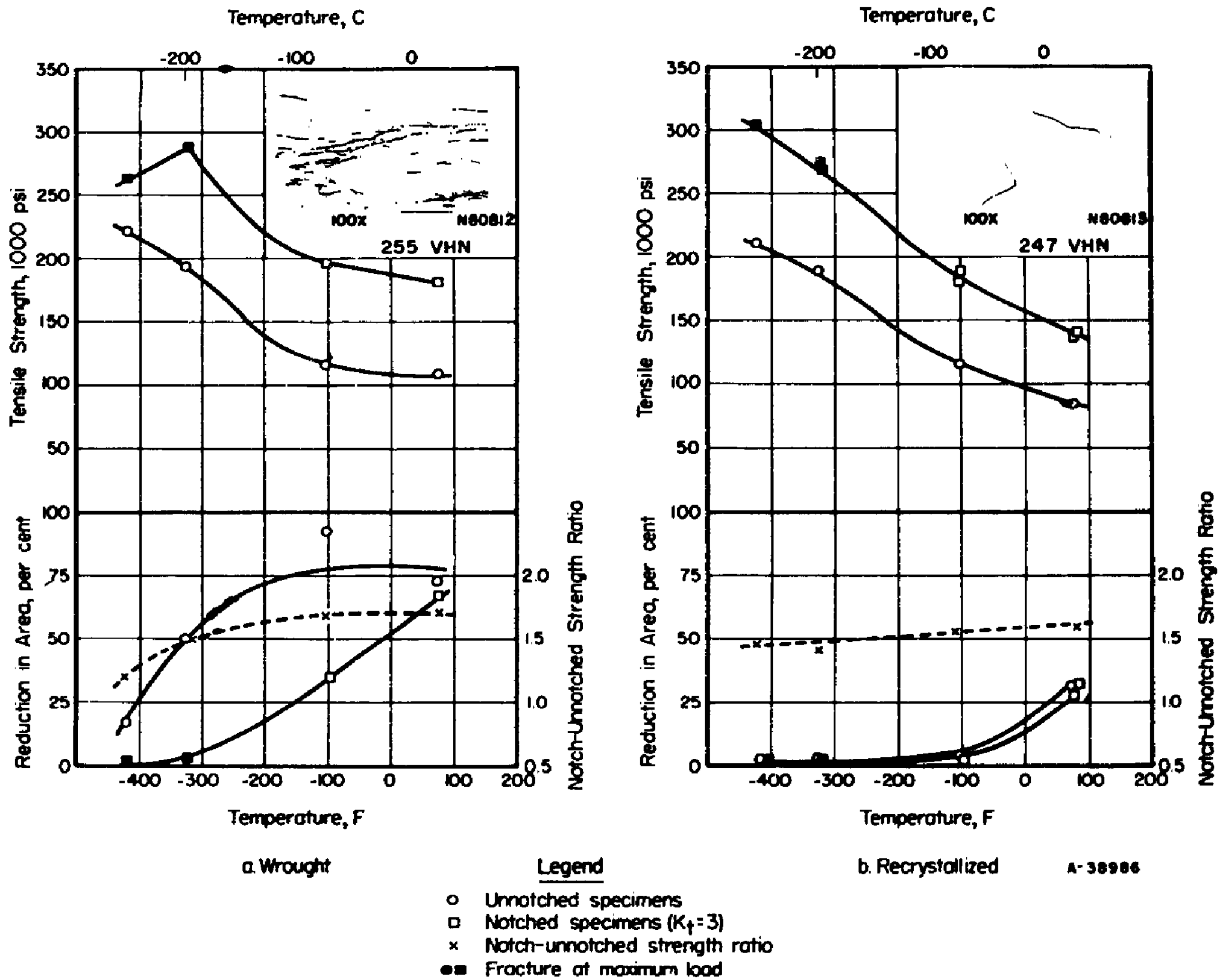


FIGURE 3. TENSILE AND NOTCH TENSILE PROPERTIES OF TANTALUM WITH 758 PPM OXYGEN

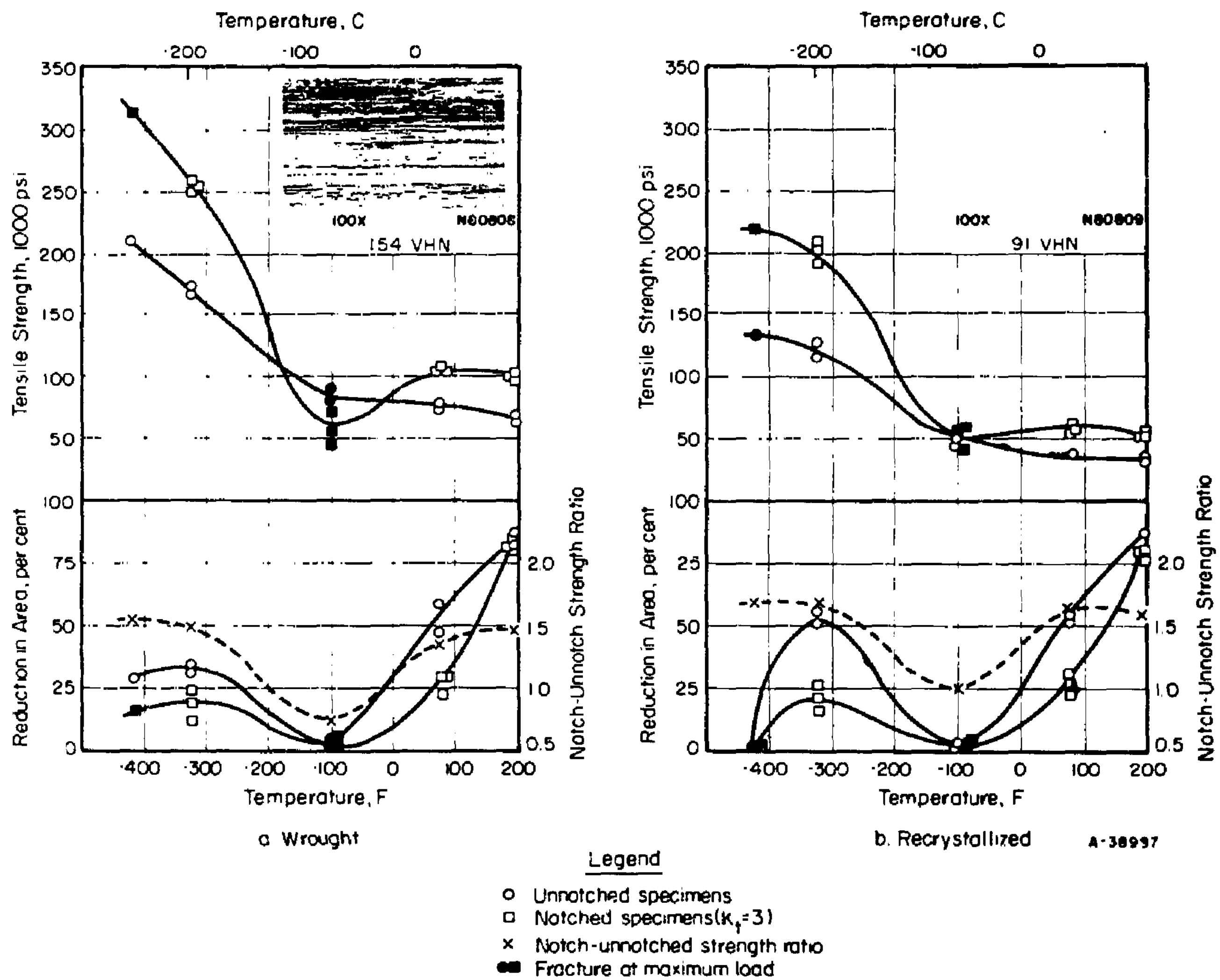


FIGURE 4. TENSILE AND NOTCH TENSILE PROPERTIES OF TANTALUM WITH 135 PPM HYDROGEN

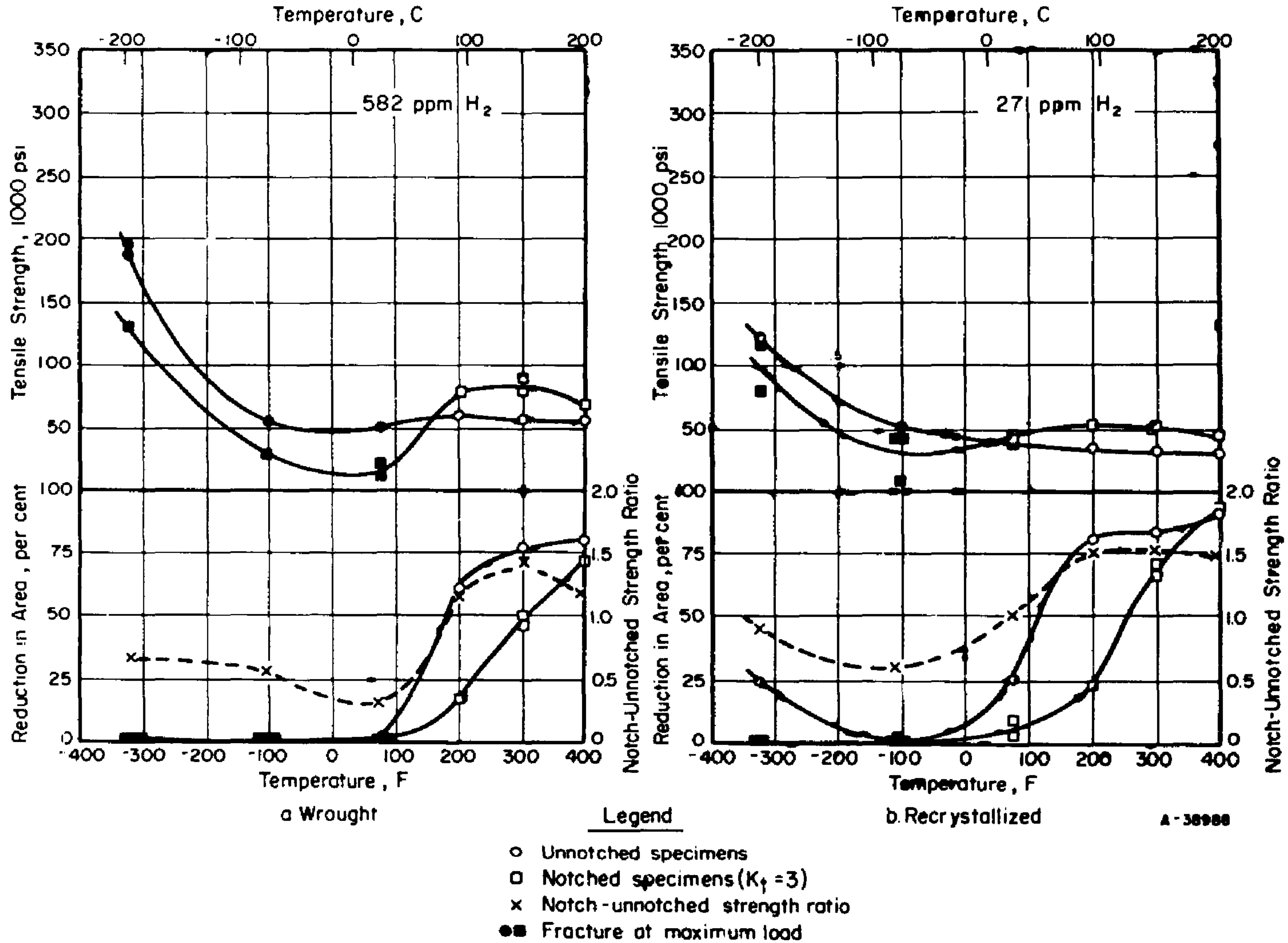


FIGURE 5. TENSILE AND NOTCH TENSILE PROPERTIES OF TANTALUM WITH "HIGH" HYDROGEN

Recrystallized columbium was notch sensitive at -420 F, whereas wrought material did not become notch sensitive at temperatures down to -420 F.

The tensile and notch tensile properties of oxygenated columbium between 75 and -420 F are illustrated in Figures 6 and 7. Oxygen moderately increased the unnotched ductility transition temperatures, but markedly increased the notched ductility transition temperature. Pronounced notch sensitivity was detected at low test temperatures. The adverse effects of oxygen were more pronounced with recrystallized structures than for wrought material, particularly at the "high" oxygen level.

Figures 8 and 9 illustrate the tensile and notch tensile properties of hydrogenated columbium. Similar to effects observed with hydrogenated tantalum, evidence of strain aging was detected at -105 F. Notch properties at -105 F were generally inferior to those at both higher and lower temperatures. Ductilities of all specimens tested at 75 F and below were negligible.

Table 6 summarizes the effects of interstitial oxygen and hydrogen on the transition characteristics and notch sensitivity of tantalum and columbium.

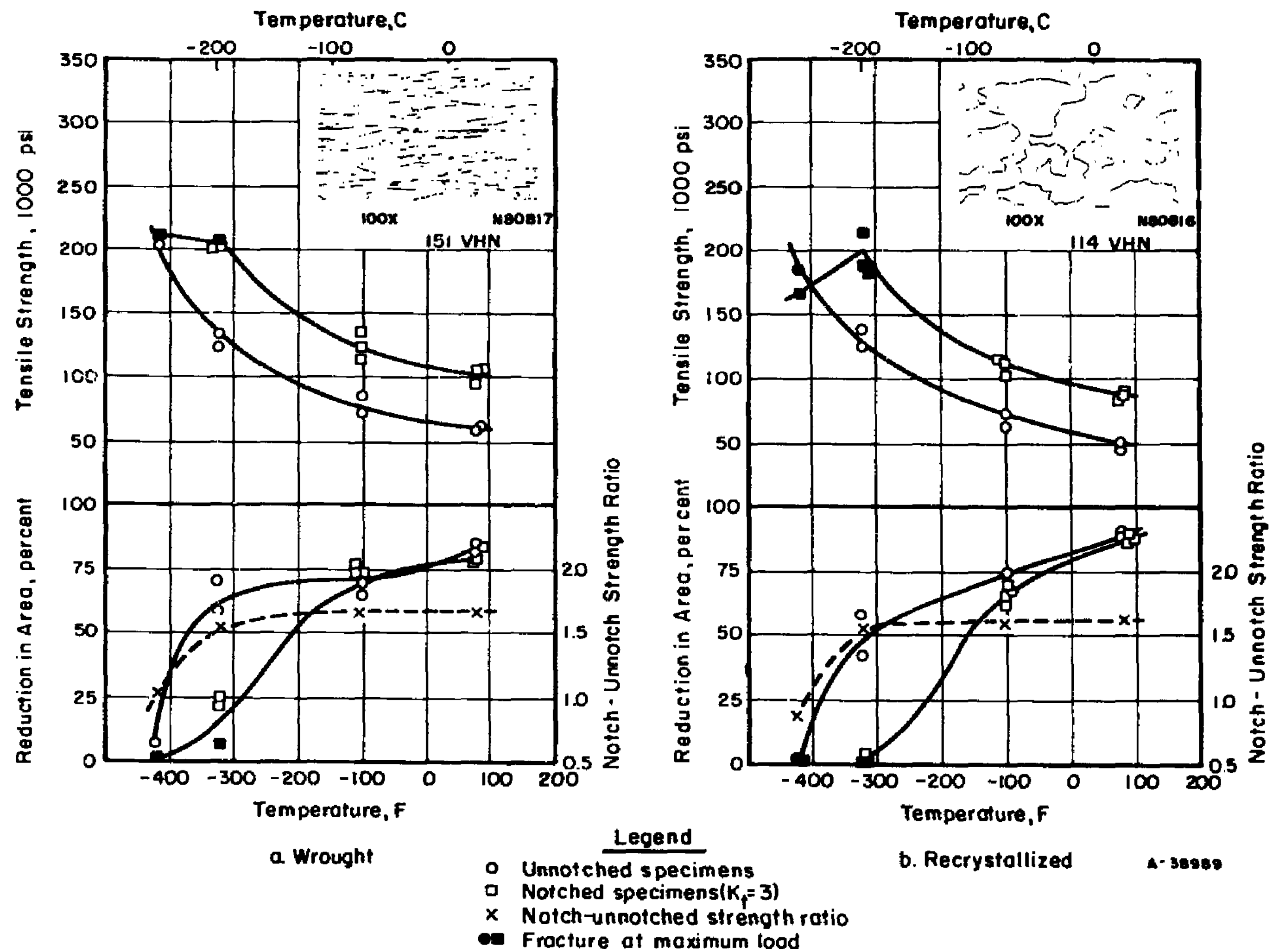


FIGURE 6. TENSILE AND NOTCH TENSILE PROPERTIES OF COLUMBIUM WITH 484 PPM OXYGEN

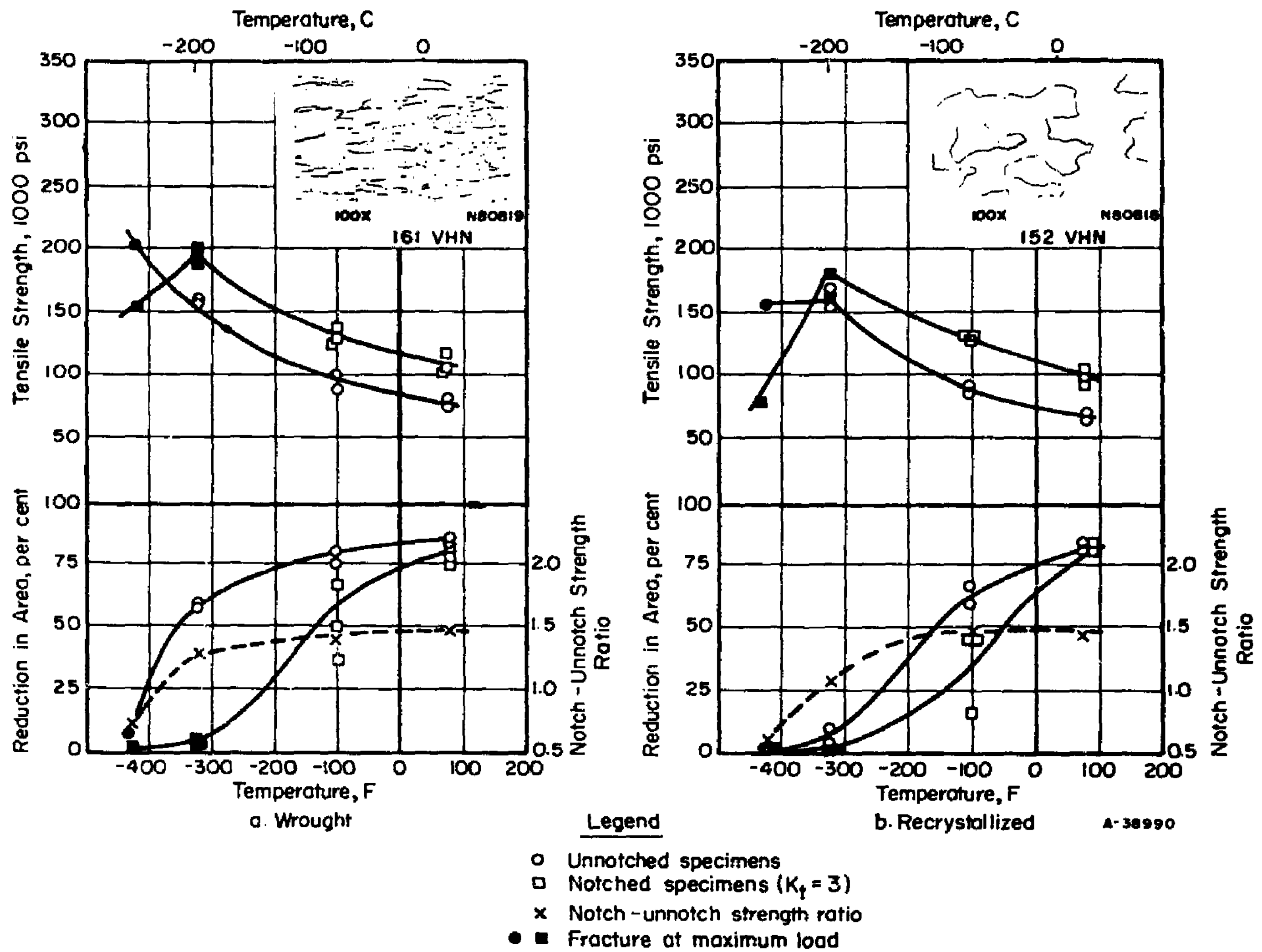


FIGURE 7. TENSILE AND NOTCH TENSILE PROPERTIES OF COLUMBIUM WITH 1320 PPM OXYGEN

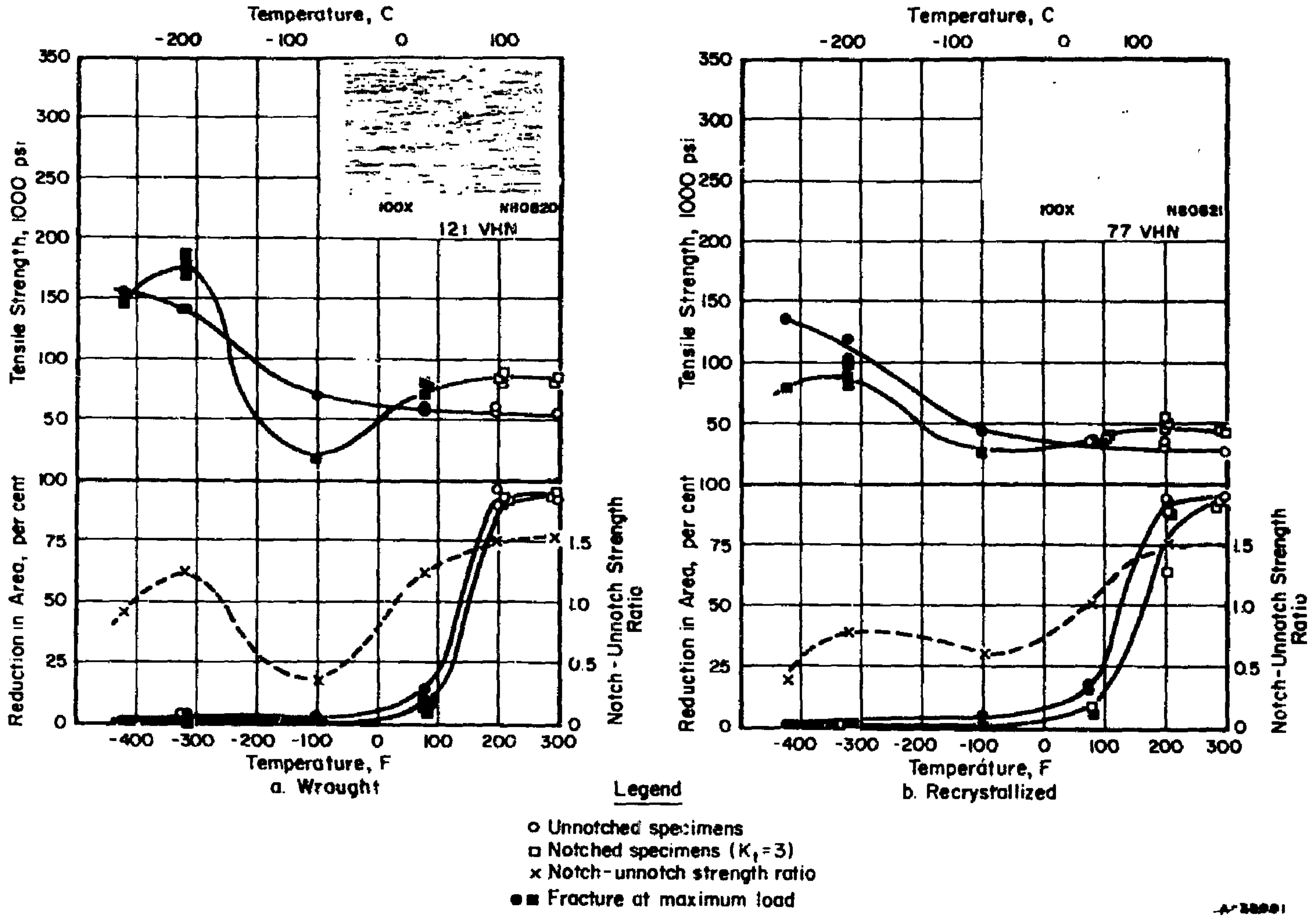


FIGURE 8. TENSILE AND NOTCH TENSILE PROPERTIES OF COLUMBIUM WITH 200 PPM HYDROGEN

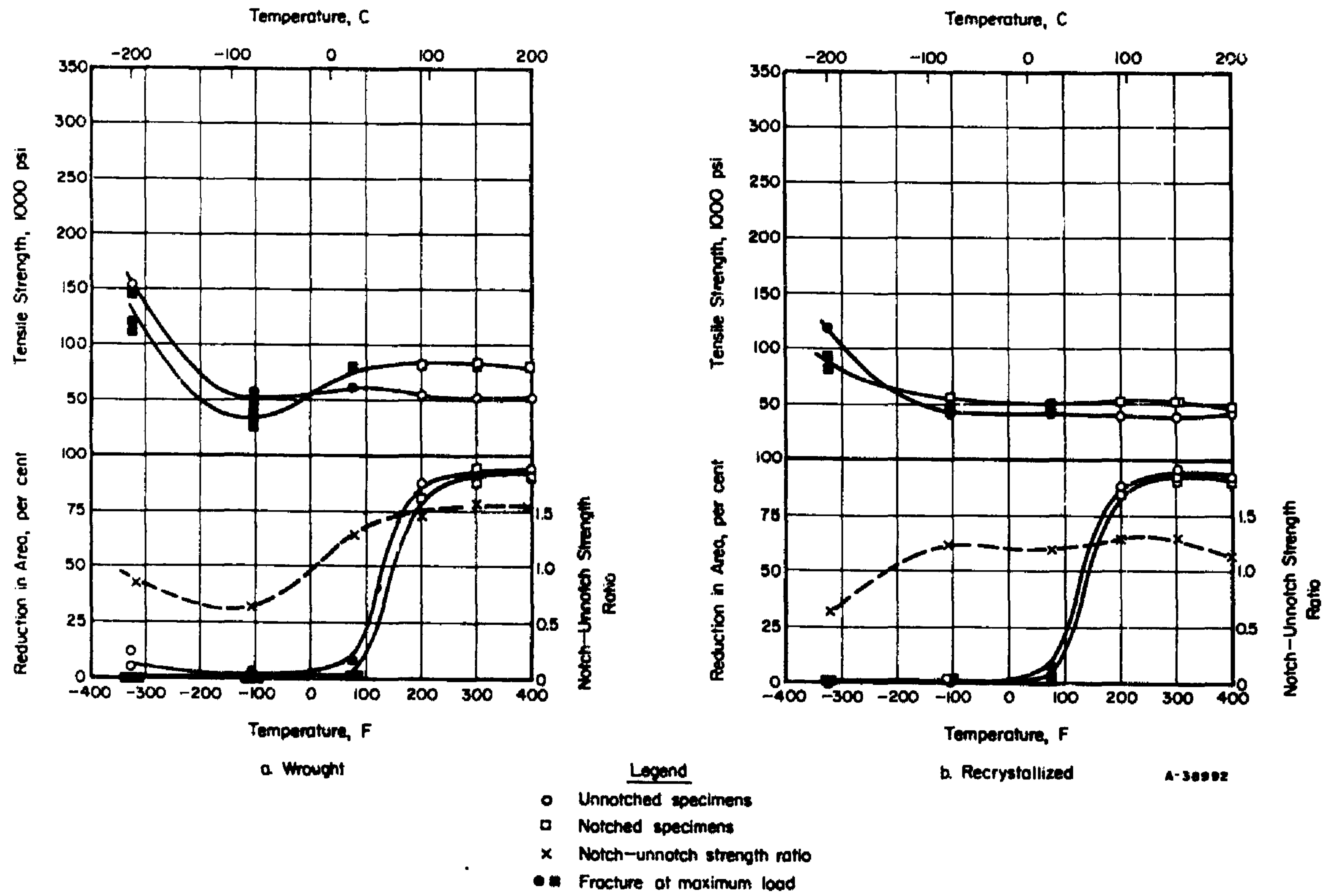


FIGURE 9. TENSILE AND NOTCH TENSILE PROPERTIES OF COLUMBIUM WITH 390 PPM HYDROGEN

TABLE 6. EFFECT OF OXYGEN AND HYDROGEN ON THE TRANSITION CHARACTERISTICS AND NOTCH SENSITIVITY OF TANTALUM AND COLUMBIUM

Interstitial Content, ppm	Condition	Ductility Transition Temperature, F: Unnotch	Ductility Transition Temperature, F: Notch	Notch Sensitivity Temperature, F
		Tantalum		
Pure[1]	Wrought	(a)	(a)	(a)
	Recrystallized	(a)	(a)	(a)
489 O_2	Wrought	-420	-360	(a)
	Recrystallized	-430	-340	(a)
758 O_2	Wrought	-350	-100	-300
	Recrystallized	50	75	(a)
135 H_2	Wrought	(b)	(b)	(-105)[c]
	Recrystallized	(b)	(b)	(-105)
582 H_2	Wrought	(b)	(b)	200
(271 H_2)	Recrystallized	(b)	(b)	200
		Columbium		
Pure[1]	Wrought	-400	-360	(a)
	Recrystallized	-390	-350	-320
484 O_2	Wrought	-390	-240	-300
	Recrystallized	-330	-190	-290
1320 O_2	Wrought	-370	-180	-300
	Recrystallized	-180	-110	-240
200 H_2	Wrought	130	--	(105) -350
	Recrystallized	130	--	200
390 H_2	Wrought	140	--	75
	Recrystallized	140	--	(d)

(a) Not observed at temperatures as low as -420 F.
(b) Obscured by strain-aging phenomena.
(c) Parentheses indicate discrete value.
(d) Data inclusive.

EFFECTS OF INTERSTITIAL ELEMENTS ON THE MECHANICAL PROPERTIES OF TANTALUM AND COLUMBIUM

Although this research program was designed specifically to determine the effects of interstitial oxygen and hydrogen in the notch behavior of tantalum and columbium, much additional data, related to the effects of interstitial impurities on mechanical properties in general, were obtained as a matter of procedure. These data are included and analyzed in an effort to develop a more complete understanding of the effects of oxygen and hydrogen on the mechanical behavior of tantalum and columbium.

Effects of Oxygen on Tantalum and Columbium

Yield Strength

The effect of oxygen content on the yield strength of tantalum and columbium at various temperatures is illustrated in Figure 10. Increasing the oxygen content of wrought and recrystallized structures increased the yield strength of both materials. This effect was somewhat more pronounced for tantalum than for columbium, but this difference is diminished when the atom per cent additions are considered. The degree of strengthening attributable to oxygen appeared to be relatively independent of temperature. The low yield strength of wrought and recrystallized columbium with high oxygen contents at -420 F is characteristic of brittle behavior.

Although for the pure columbium the yield strength of the wrought condition was about 20,000 psi greater than for the recrystallized structure, this difference narrowed to about 5,000psi at the high oxygen level. This is similar to the tensile strength behavior reported earlier. It is shown in Appendix I that oxygen does not significantly affect the work-hardening characteristics of columbium. Therefore, it appears that working tends to mask the strengthening effects of oxygen on columbium. Or it can be said that oxygen is a more effective strengthener of the recrystallized structure than of the wrought material. When comparing strengths at the low and high ppm oxygen level, it should be remembered that it was necessary to fabricate the materials with high oxygen content at elevated temperatures. This may have caused appreciable recovery during fabrication, resulting

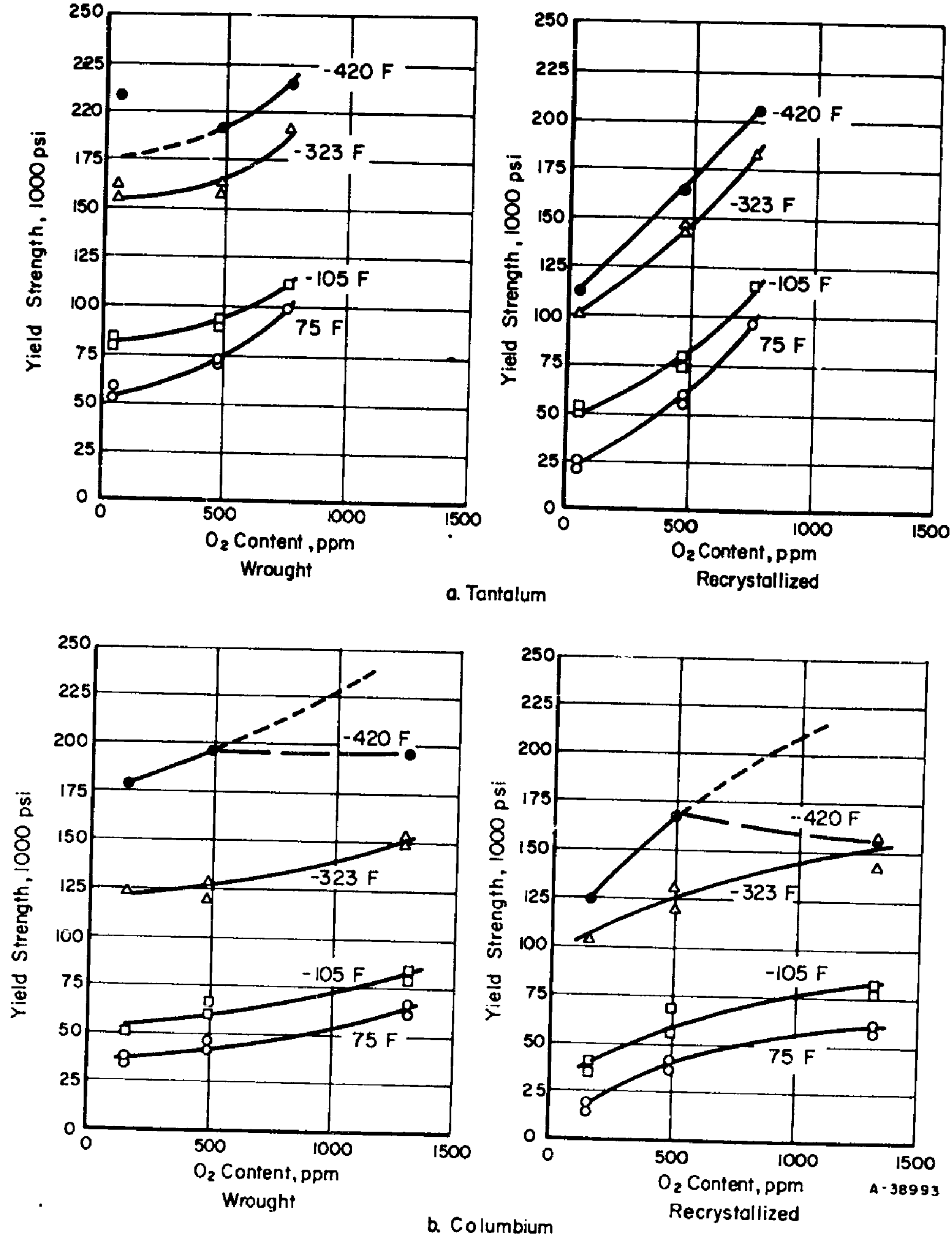


FIGURE 10. EFFECT OF OXYGEN CONTENT ON THE YIELD STRENGTH OF TANTALUM AND COLUMBIUM

in lower-than-expected strengths for the wrought material with high oxygen contents. Similar behavior was observed for tantalum, but was less pronounced.

Ductility

Figure 11 illustrates the effect of oxygen on the unnotched ductility of tantalum and columbium at several temperatures. In general, increasing the oxygen content decreased the reduction in area. This effect was most pronounced at the -323 and -420 F test temperatures. In addition, ductilities at all test temperatures were equal or lower for both materials with recrystallized structures than in the wrought condition. High oxygen contents, therefore, are more detrimental to the ductility of recrystallized tantalum and columbium than to wrought material.

Increasing the oxygen content of tantalum above 500 ppm reduced ductility more severely than did the initial oxygen increment. In columbium, oxygen contents above 500 ppm appeared to have a proportionately smaller effect than lower oxygen contents.

Transition Temperature

Figure 12 illustrates the effect of oxygen on the ductile-to-brittle transition behavior of tantalum and columbium. Recrystallized structures exhibited higher transition temperatures than wrought materials. Furthermore, increasing the oxygen content raised the transition temperature of the recrystallized specimens to a greater extent than was observed for the wrought materials. Therefore, in effect, the difference between the transition temperatures of recrystallized and wrought structures widened as the oxygen content was increased, and recrystallized structures became increasingly disadvantageous.

Behavioristically, this is similar to what is observed with the refractory metals in Group VIa (Cr, Mo, and W). The embrittlement of these metals is generally associated with the segregation of impurity atoms at grain boundaries and/or the detrimental effects of a large grain size. Metallographic examination, as described in Appendix I, did not reveal the presence of a massive-grain-boundary second phase or a pronounced tendency toward intergranular fracture in either tantalum or columbium. The apparent lack of intergranular cracks in tantalum and columbium suggests that different mechanisms of embrittlement are operative in Group Va and Group VIa.

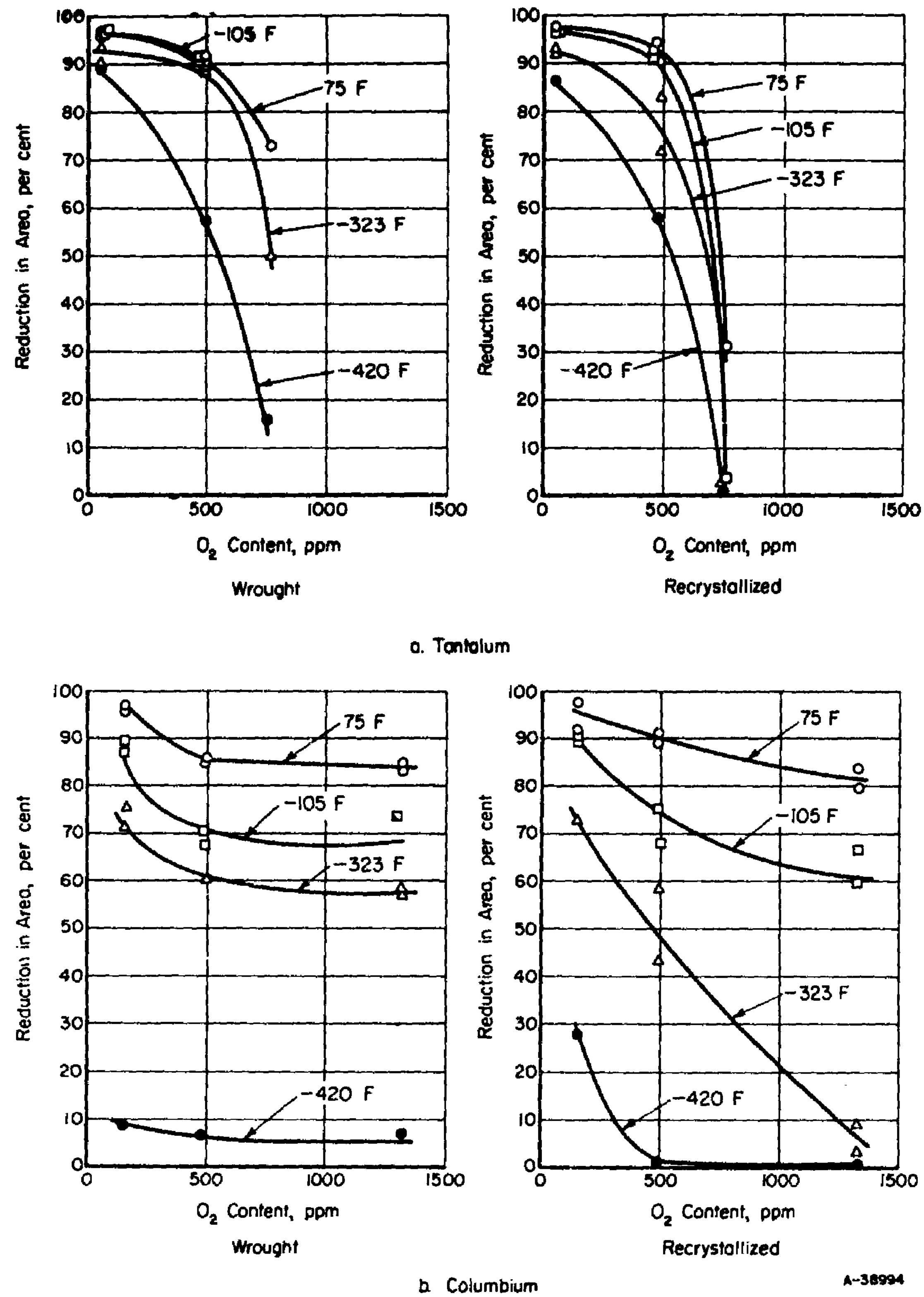


FIGURE 11. EFFECT OF OXYGEN CONTENT ON THE UNNOTCHED DUCTILITY OF TANTALUM AND COLUMBIUM

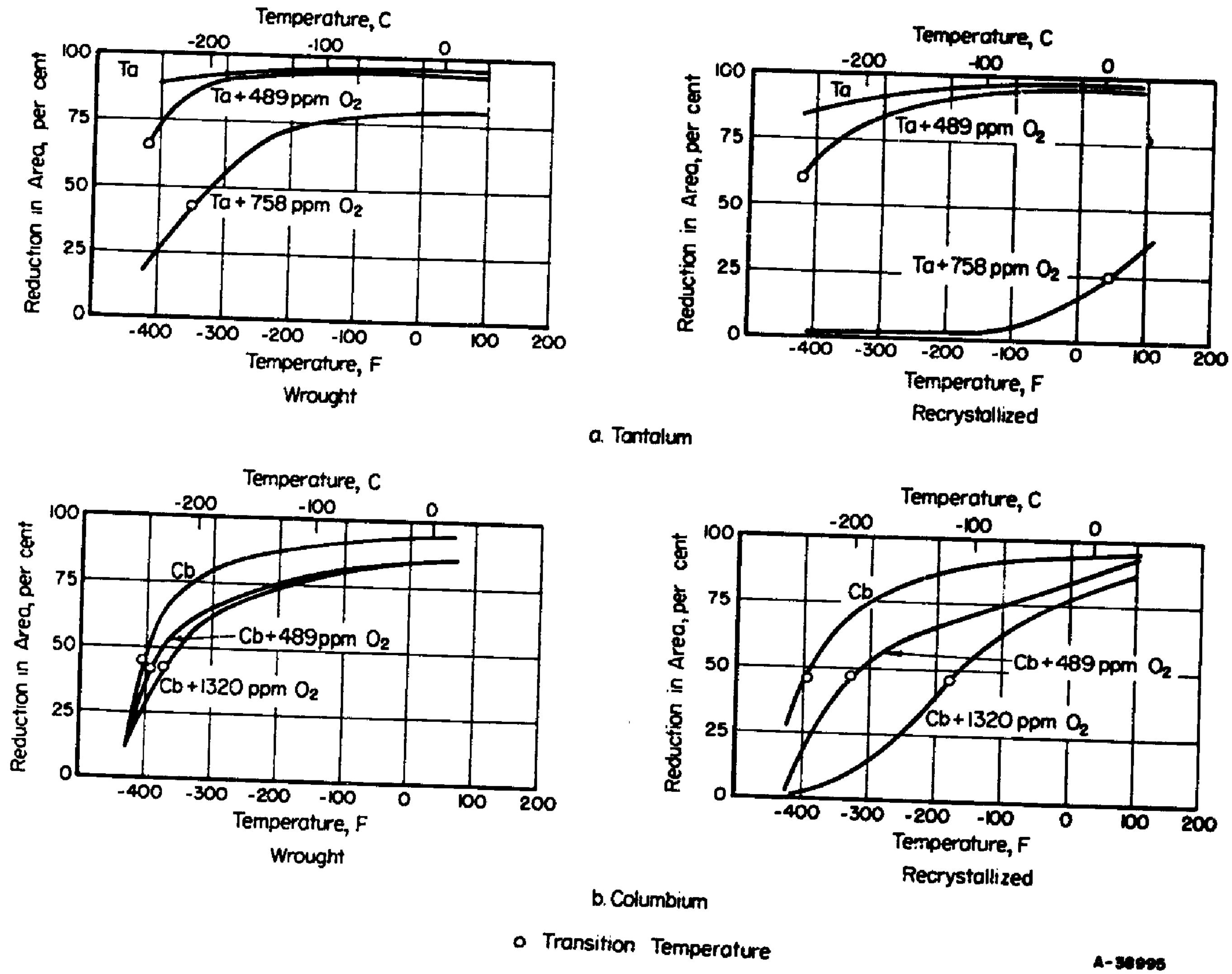


FIGURE 12. EFFECT OF OXYGEN ON THE DUCTILE-TO-BRITTLE TRANSITION OF TANTALUM AND COLUMBIUM

Notch Sensitivity

Figure 13 summarizes the effect of oxygen on the notch-unnotch strength ratio of tantalum and columbium. In general, it is shown that increasing the oxygen content in tantalum and columbium contributes to inferior notch behavior. Particularly for columbium, the deleterious effects of oxygen tend to be more pronounced for recrystallized structures than for wrought material.

A comparison of Figure 12 with Figure 13 for each material and condition, indicates that in general inferior notch behavior occurs at temperatures somewhat below those at which unnotch ductility begins to deteriorate (the upper limit of the ductile-to-brittle transition temperature range). This should be expected since notch sensitivity is usually the result of low ductility and the inability of the material to flow and relieve stress concentrations. An exception is recrystallized tantalum. Ductility at approximately -100 F and below is marginal, but no evidence of notch sensitivity was detected at temperatures as low as -420 F.

Effects of Hydrogen on Tantalum and Columbium

Yield Strength

Figure 14 illustrates the effect of hydrogen on the yield strength of tantalum and columbium. Hydrogen increases the yield strength slightly of both wrought and recrystallized structures of these materials. At the higher hydrogen levels, many of the specimens failed before yielding or with very low ductilities. Therefore, they exhibited relatively low apparent yield strengths. In general, the strengthening effect of hydrogen appears to be nearly uniform over the range of hydrogen contents investigated, and was not appreciably altered by test temperature.

Ductility

The effect of hydrogen on the ductility of tantalum and columbium is illustrated in Figure 15. Although, hydrogen had only a moderate effect on the strength of wrought and recrystallized tantalum, it can be seen that hydrogen additions severely reduced the ductility of tantalum and columbium. This effect was more pronounced for columbium than for tantalum. Hydrogen additions in columbium beyond 150 ppm had a proportionately smaller effect than additions up to 150 ppm. Wrought and recrystallized structures showed approximately the same sensitivity to hydrogen additions.

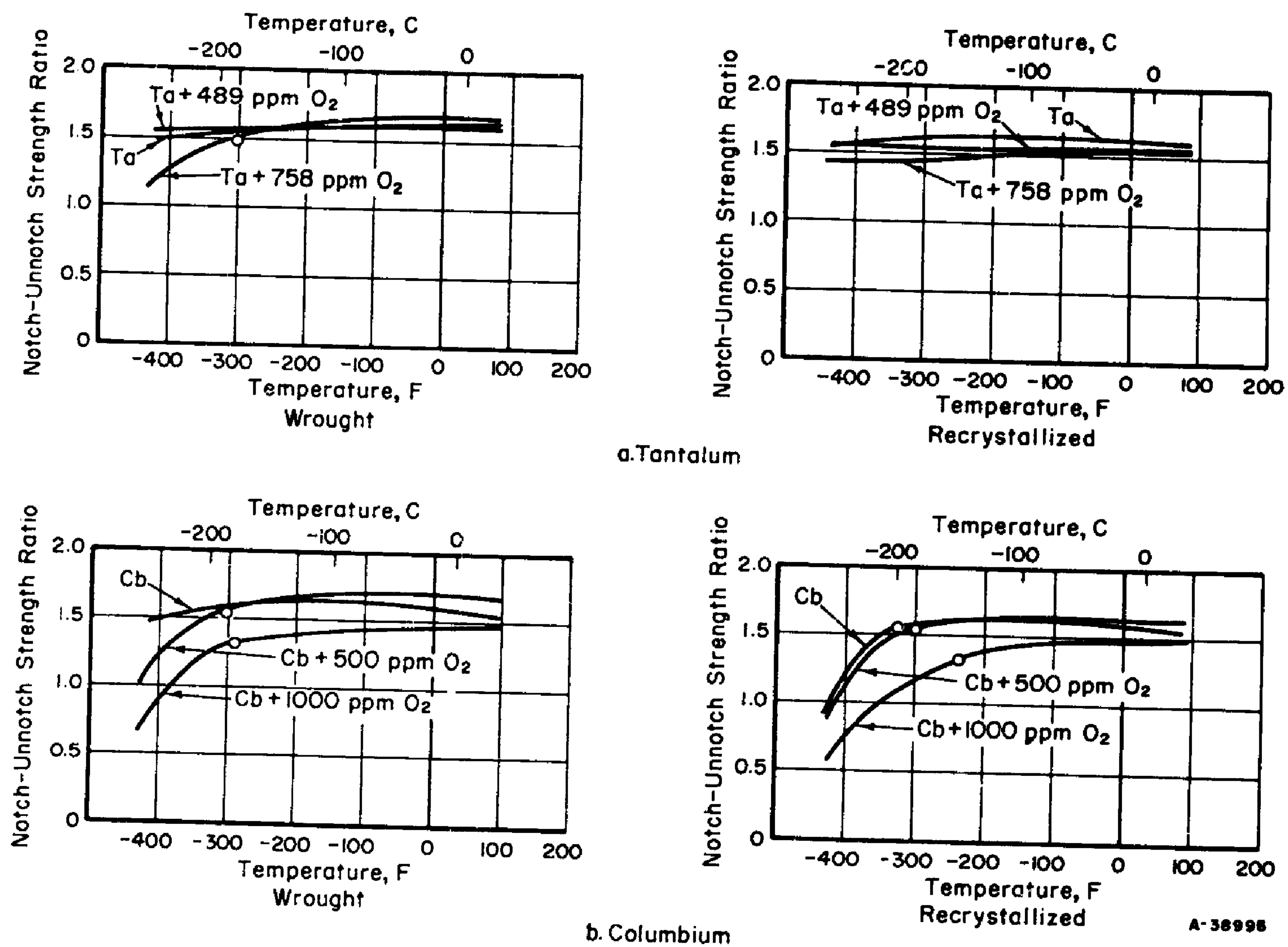


FIGURE 13. EFFECT OF OXYGEN ON THE NOTCH-UNNOTCH STRENGTH RATIO OF TANTALUM AND COLUMBIUM

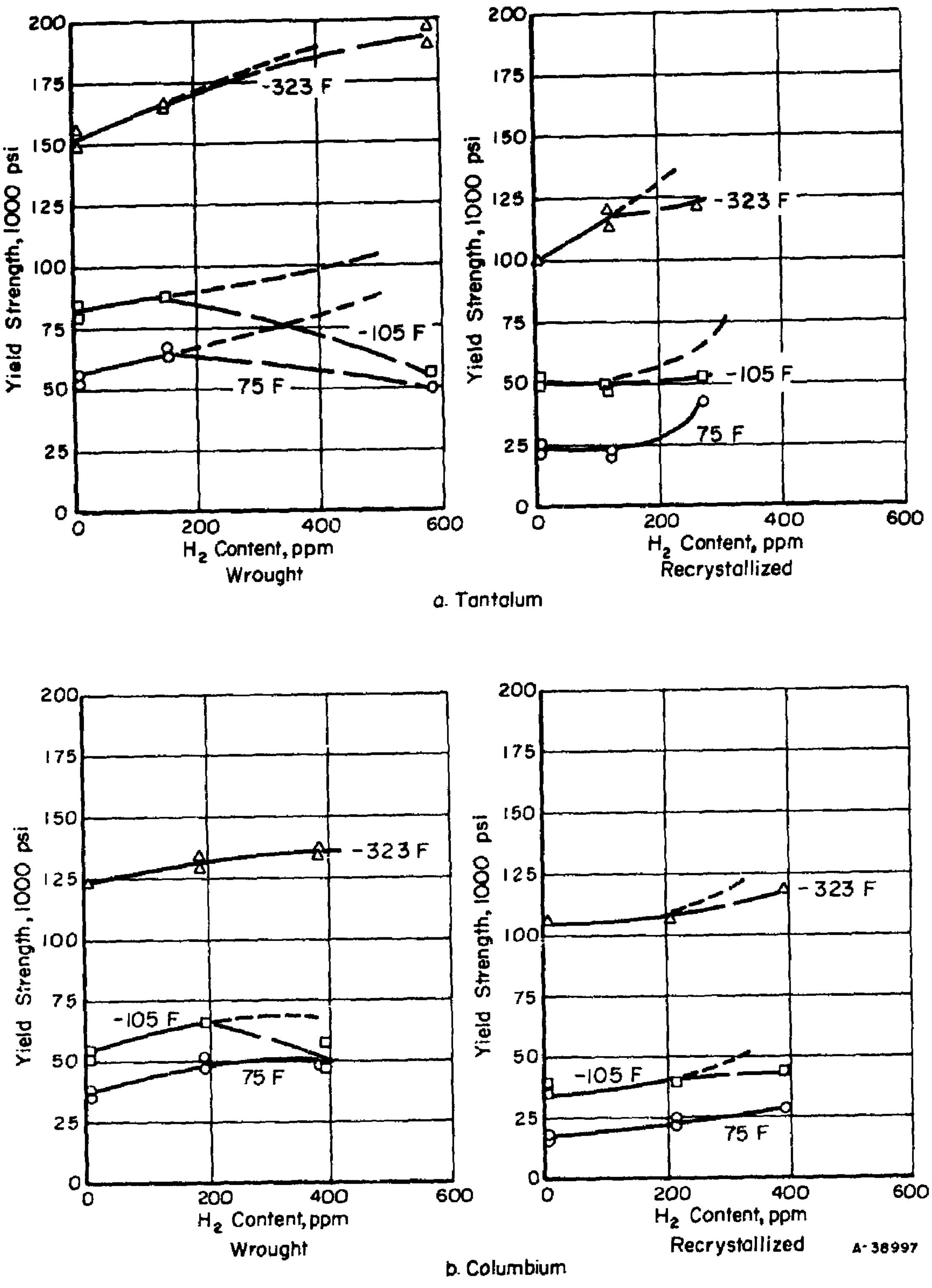
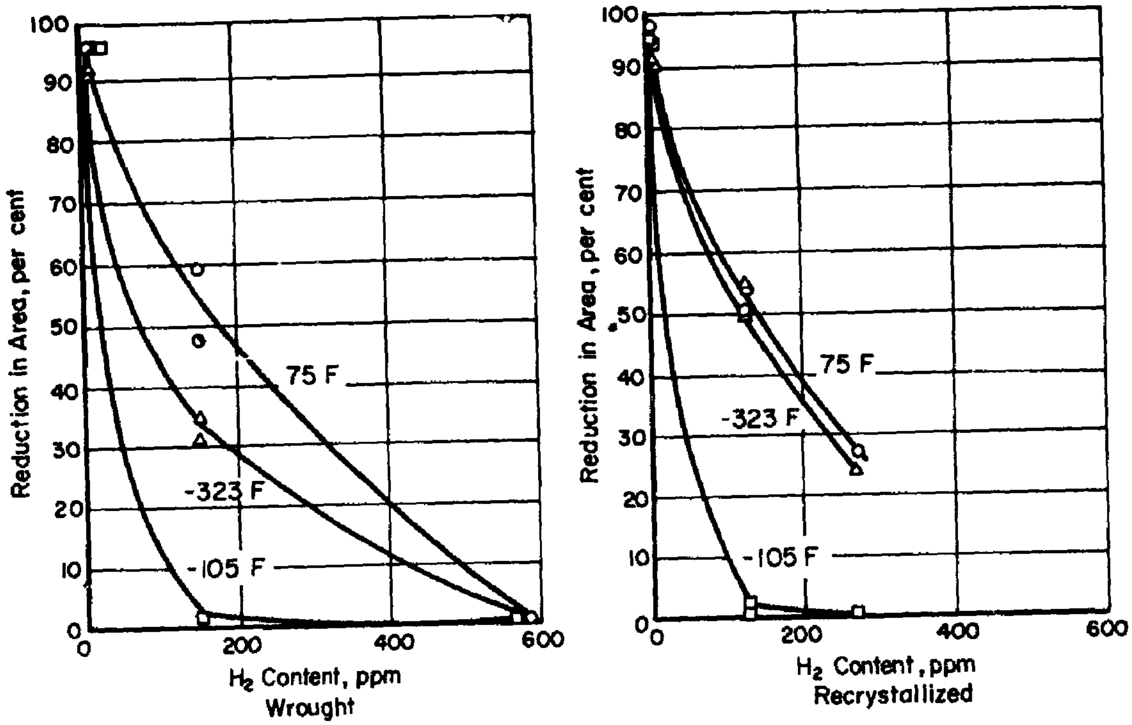
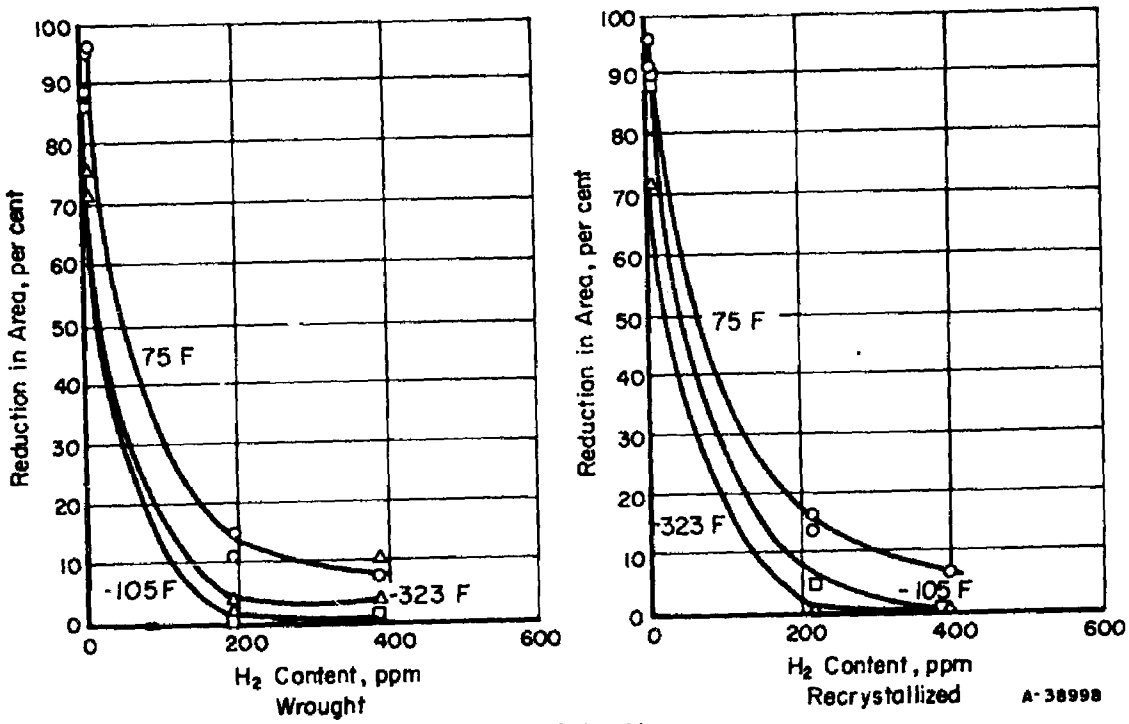


FIGURE 14. EFFECT OF HYDROGEN CONTENT ON THE YIELD STRENGTH OF TANTALUM AND COLUMBIUM



a. Tantalum



b. Columbium

FIGURE 15. EFFECT OF HYDROGEN CONTENT ON THE DUCTILITY OF TANTALUM AND COLUMBIUM

As previously noted, ductilities at -105 F were low but ductilities at both higher and lower temperatures were greater. Minimum points in reduction in area versus temperature curves, such as indicated for tantalum and columbium at -105 F, are an indication of strain aging[2]. Cottrell[3] has suggested that strain aging is a diffusion-controlled phenomenon and that the maximum effects of strain aging should occur when

$$D = 10^{-9} \dot{\epsilon}$$

where $\dot{\epsilon}$ is the strain rate and D is the diffusion rate. D can be expressed as

$$D = Do \exp \frac{-Q}{RT} ,$$

where Do is a constant, Q is an activation energy, R is the gas constant, and T is the absolute temperature. Therefore, by using these two equations, it should be possible to calculate the temperature at which strain-aging effects due to a particular interstitial element are a maximum. The values used, and calculated temperatures for maximum strain-aging effects are as follows:

Parameter	Tantalum	Columbium
Q	-29,460[a]	-9,370[b]
D_o	305[a]	0.0214[b]
T calc.	390 F	-60 F

(a) From the present research.
(b) DMIC Memorandum 50.

This relationship predicts the minimum points in reduction in area for hydrogenated columbium with reasonable accuracy, but does not fit the observed data for hydrogenated tantalum nearly as well.

Transition Temperature

The effect of hydrogen on the ductility transition of tantalum and columbium is illustrated in Figure 16. Minimum points were observed in the reduction in area versus temperature curves of tantalum at -105 F. As previously explained, this could be the result of a strain-aging phenomenon. The normal ductility transition appears to be masked by this behavior, and little can be concluded about the effect of hydrogen on the ductility transition temperature of tantalum as such. However, it is obvious that hydrogen additions severely reduced the ductility of tantalum below approximately 200 F. Behavior of wrought and recrystallized materials were comparable.

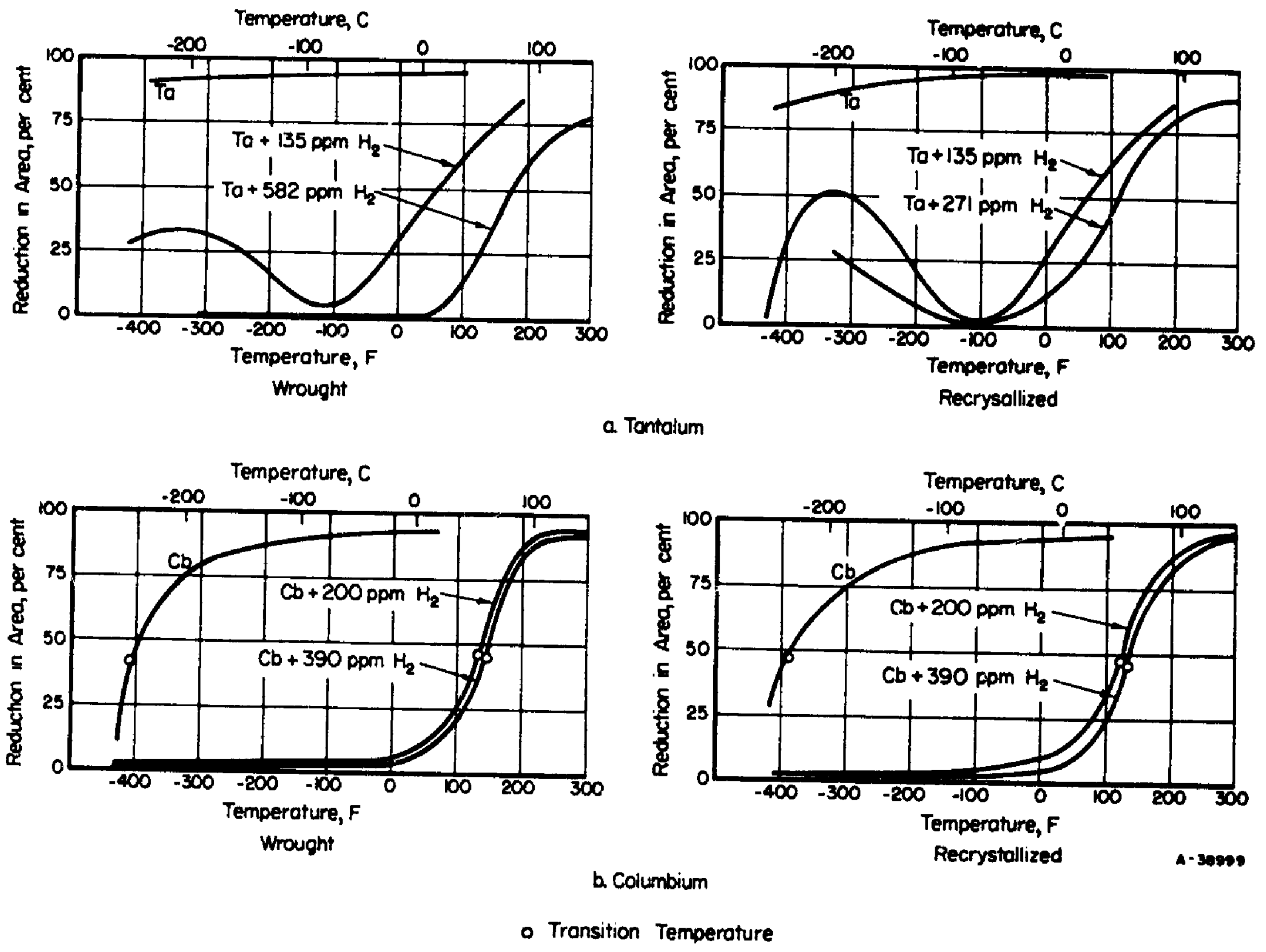


FIGURE 16. EFFECT OF HYDROGEN ON THE DUCTILE-TO-BRITTLE TRANSITION OF TANTALUM AND COLUMBIUM

Additions of 200 ppm hydrogen to columbium markedly raised its effective transition temperature, but further increasing the hydrogen content to 390 ppm did not appreciably alter the transition temperature, or reduce the ductility. Indications of strain-aging in the ductility curves were not as apparent as was observed with tantalum. The effect of strain-aging on transition characteristics is not clear. There was no appreciable difference between wrought and recrystallized structures.

Notch Sensitivity

Figure 17 illustrates the effect of hydrogen on the notch-unnotch strength ratio of tantalum and columbium. The most notable feature of the notch-unnotch strength ratio curves for hydrogenated tantalum and columbium is the minimum points generally in the vicinity of - 105 F. The poor notch properties at this temperature are thought to be a result of low ductilities due to a strain-aging phenomenon caused by the interaction of hydrogen atoms with dislocations as previously described. If this is actually a strain-aging phenomena, it can be speculated that inferior notch behavior may result at temperatures where the interaction of other interstitial atoms (carbon, oxygen, nitrogen) with dislocations is a maximum. This matter bears further examination.

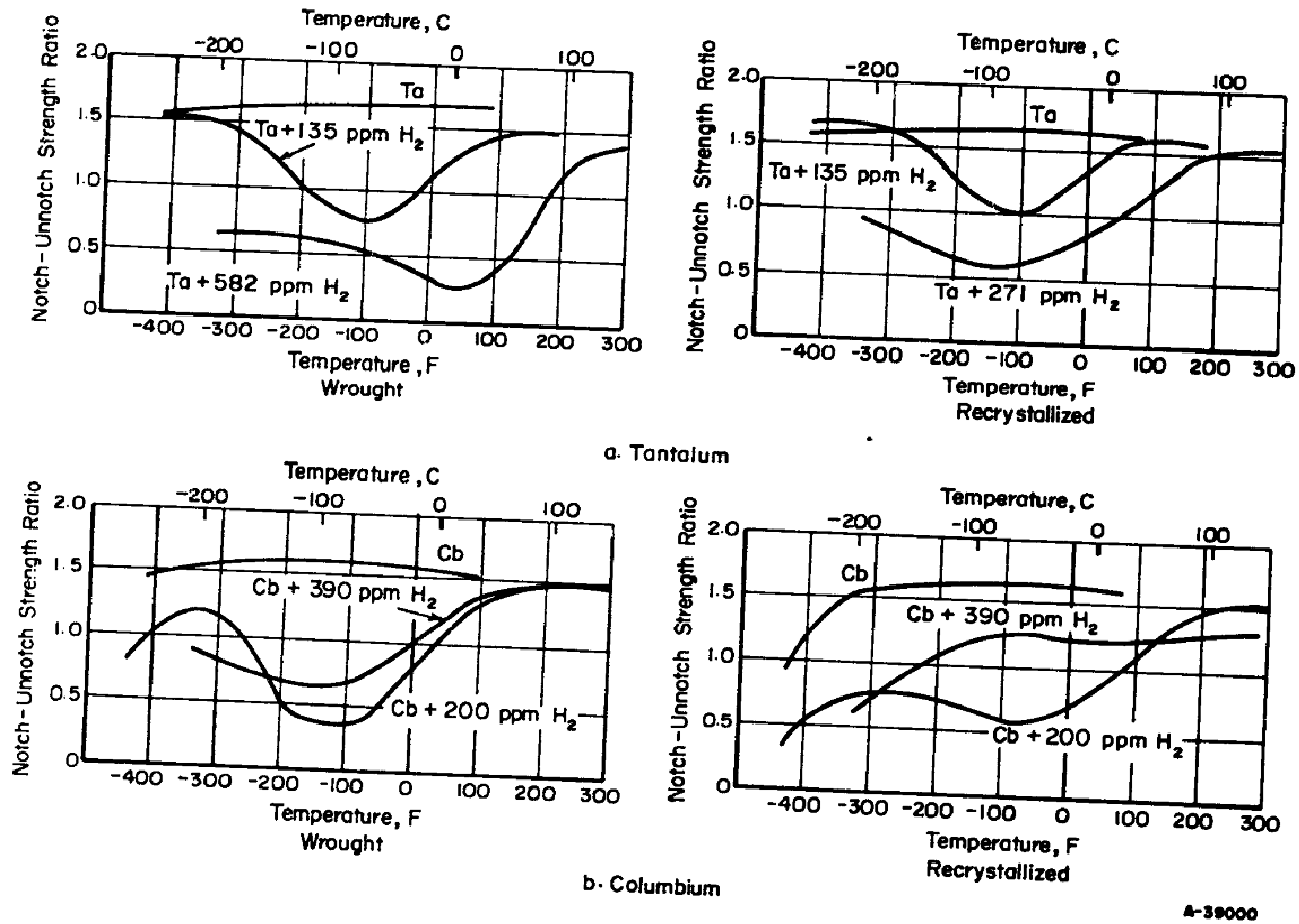


FIGURE 17. EFFECT OF HYDROGEN ON THE NOTCH-UNNOTCH STRENGTH RATIO OF TANTALUM AND COLUMBIUM

COMMERCIAL ALLOYS

Ta-10W Tantalum Alloy

Ta-10W is a moderately high strength tantalum alloy designed for structural service at elevated temperatures.

Figure 18 illustrates the tensile and notch tensile properties of wrought and recrystallized Ta-10W between -420 and 75 F. Grain size of the recrystallized specimens was considerably finer and more uniform than that of the recrystallized tantalum.

Trends toward low ductility were observed at low test temperatures and were somewhat more pronounced for recrystallized structures than for wrought material. Appreciable ductility was still retained at -420 F in all conditions. No indication of notch sensitivity was observed even at -420 F.

F-48 Columbium Alloy

F-48 columbium alloy (Cb-15W-5Mo-1Zr-0.03C) was developed by the General Electric Company for use as a structural material having high strength (particularly in creep exposures) in the temperature range 2000 to 2500 F. Its high strength is due to the tungsten and molybdenum solid-solution additions and to a dispersed carbide second phase.

The tensile and notch tensile behavior of wrought and recrystallized F-48 are illustrated in Figure 19. Recrystallized F-48 exhibited a fine, uniform grain size. A very fine, uniformly distributed second phase was evident in both the wrought and recrystallized structures. The lack of softening normally expected during recrystallization has been attributed to partial solutioning of the second phase followed by reprecipitation during cooling from the heat-treating temperatures[(2)]. Despite the lack of softening, loss in strength was observed as a result of recrystallization.

Wrought and recrystallized notched specimens exhibited negligible ductilities at all test temperatures below 300 F. Considerable scatter in the notched strength values, particularly of wrought material, suggests unreliable notch behavior for F-48 at temperatures below 300 F. Transition temperature was around 0 F for the wrought condition, and above room temperature for recrystallized material.

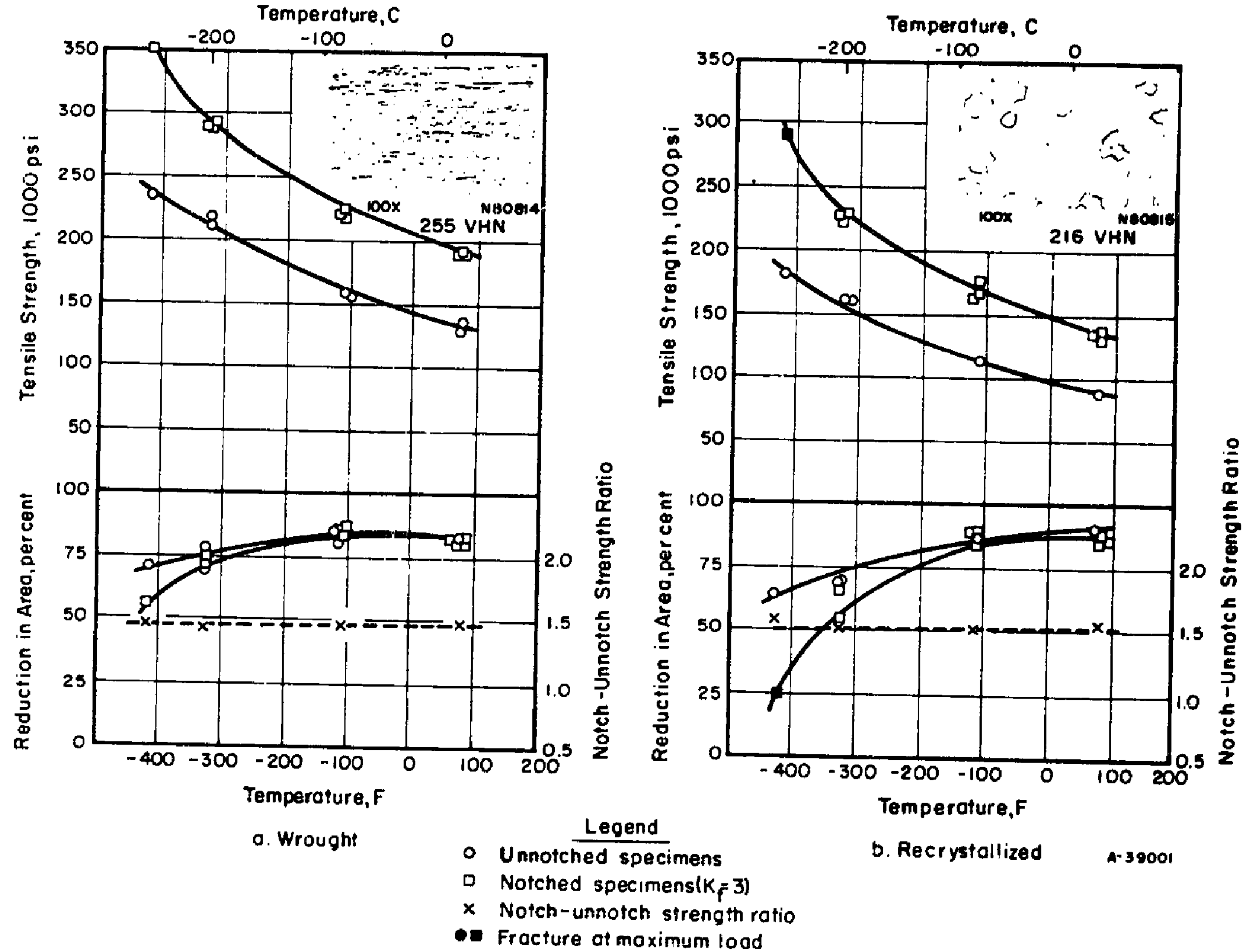


FIGURE 18. TENSILE AND NOTCH TENSILE PROPERTIES OF Ta-10W

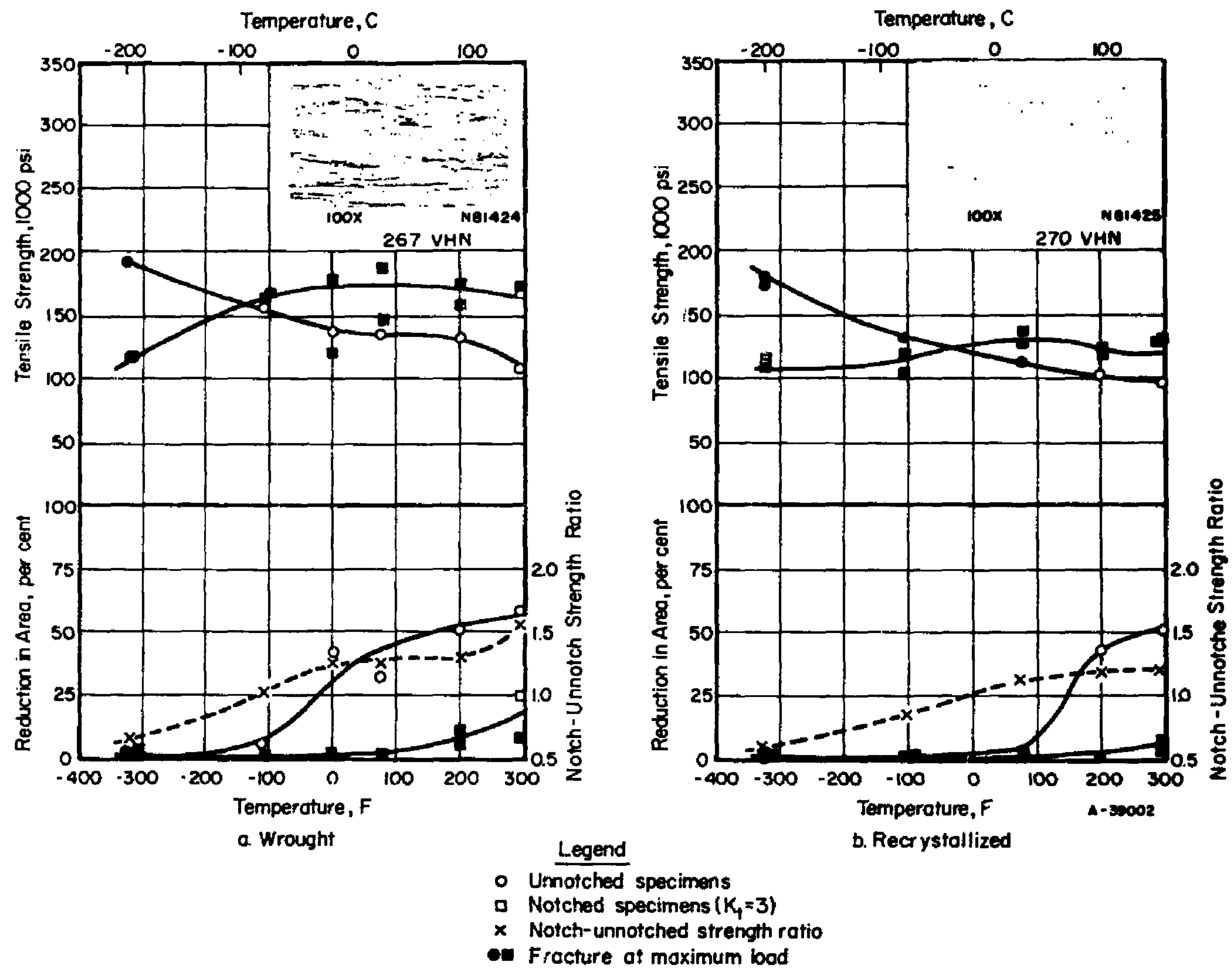


FIGURE 19. TENSILE AND NOTCH TENSILE PROPERTIES OF F-48 COLUMBIUM ALLOY

DISCUSSION

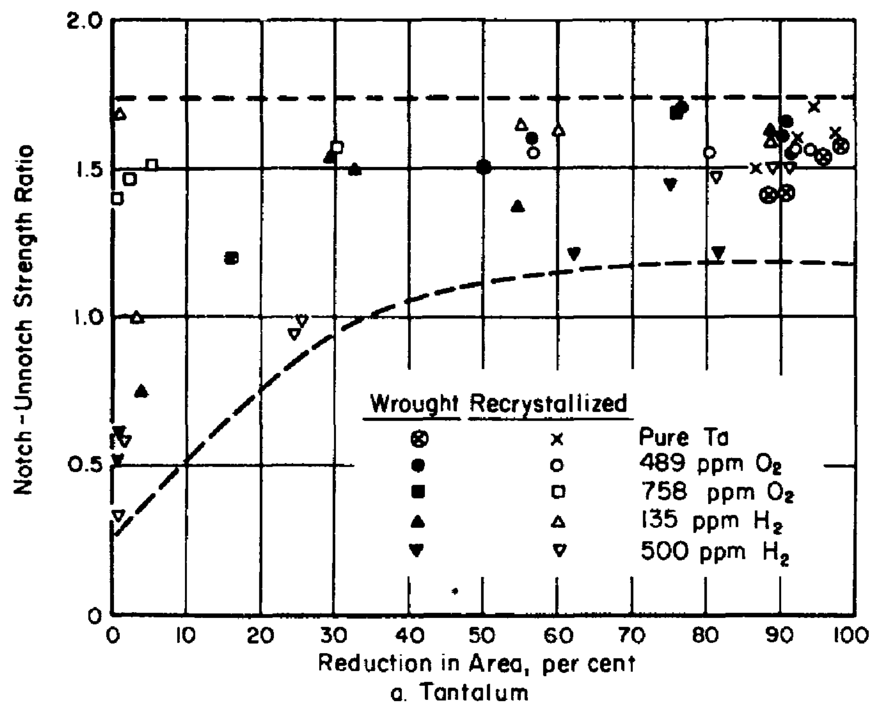
High oxygen contents in tantalum and columbium increased the strength, lowered the ductility at given temperatures, raised the ductility transition temperature and resulted in inferior notched properties. The deleterious effects of oxygen were more pronounced for recrystallized specimens than for wrought material.

Hydrogen additions increased the strength of tantalum and columbium slightly and markedly reduced their ductility. Unusual behavior at -105 F, thought to be a strain-aging phenomenon due to the interaction of hydrogen atoms with dislocations, generally resulted in low ductilities and poor notch behavior.

The data indicate that high oxygen and hydrogen contents do cause brittle behavior and poor notch behavior. Therefore, if these materials are used in applications where brittle failure could be a serious problem (i.e., at low temperatures or under multiaxial stresses) care should be taken to prevent contamination either during fabrication or in service at elevated temperatures.

It is desirable to get some indication of notch behavior from data derived from a simple, unnotched tensile test. Poor notch properties are generally associated with materials having low ductility under certain conditions of temperature and purity. If a material cannot flow plastically and relieve stress concentrations at the base of a notch, it is likely that cleavage cracks will form in the stress concentrations, and propagate at lower nominal stresses than are obtained with unnotched specimens. An attempt is made in Figure 20 to establish a correlation between unnotch reduction in area and the notch-unnotch strength ratio for tantalum and columbium. It is obvious that no simple relationship exists between unnotch ductility and notch behavior. Some specimens had reductions in area as high as approximately 30 per cent and were notch sensitive, whereas others exhibited very low ductility but were not significantly notch sensitive. This suggests that some other factor besides the inherent ductility of a material must be important in determining notch behavior.

If the primary mechanism of flow and fracture is by shear, it is expected that the notch strength under simple uniaxial loading should be greater than the unnotch strength because the notch reduces the maximum effective shear stress in the test section. However, if the primary mechanism of failure is by cleavage, it could be expected that cleavage cracks will nucleate in the tensile stress concentration at the base of the notch and propagate at lower nominal stresses in notched specimens than in unnotched specimens. Although exceptions can be noted, fracture mode observations



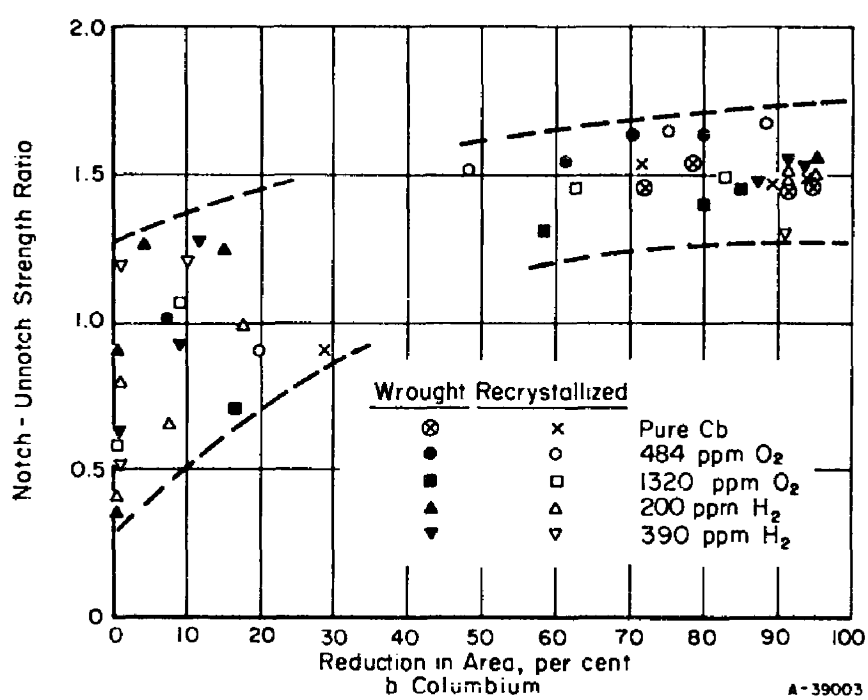


FIGURE 20. RELATIONSHIP BETWEEN UNNOTCH DUCTILITY AND NOTCH BEHAVIOR FOR TANTALUM AND COLUMBIUM

made in this research program generally confirm that when the notch-unnotch strength ratio was less than 1.0., the notched specimens failed primarily by the cleavage mechanism. This model would help explain the relatively better notch behavior of tantalum than of columbium, since it appears that cleavage crack initiation is more difficult in tantalum than in columbium. It would also help explain why hydrogenated tantalum and columbium generally exhibited poorer notch behavior than oxygenated material since hydrogen appears to be more effective in promoting cleavage fracture than does oxygen. These effects are presented and discussed in detail in Appendix I.

Ductility, then, is thought to contribute to good notch properties insofar as it allows the material to flow and relieve stress concentrations at the base of a notch. Nevertheless, if cleavage cracks still form in the stress concentration and propagate after measureable plastic flow, poor notch properties may result. The precise effect of a notch on the mechanism of fracture initiation and propagation is largely unknown and bears further investigation.

Data upon which this section of this report is based are included in Battelle Laboratory Record Book No. 15770, pages 1-100 inclusive.

PART II. REACTIONS IN THE TANTALUM-HYDROGEN SYSTEM

Manley W. Mallett
Billy G. Koehl

INTRODUCTION

To properly evaluate the effects of interstitial elements on the mechanical properties of a metal, it is useful and, in some cases, necessary to know their kinetics of sorption by and diffusion in the metal. The literature is lacking in this type of information for the behavior of hydrogen and tantalum. At the temperatures of interest, above 300 C (570 F), the tantalum-hydrogen system shows a continuous solid solution up to compositions corresponding to $TaH_{0.5}$ and higher. It has been demonstrated[5,6] that kinetic data for such a system are more readily rationalized when kinetic experiments are designed to produce a reaction product of constant composition. Therefore, it was necessary to know the equilibria of the tantalum-hydrogen system before starting the kinetic experiments. A number of earlier observations on the solubility of hydrogen in tantalum have been made.[7] However, all of the data, with exception of those of Sieverts and Bergner and Sieverts and Bruning, appear to suffer from the impurity of the tantalum. The results show gross effects presumably of dissolved or surface oxide inhibiting reaction and quantitatively affecting the solution. Sieverts equilibria were too limited for our purpose since they are largely for a pressure of 760 mm of mercury.

A recent paper[8] by P. Kofstad, W. E. Wallace, and L. J. Hyvönen gave good equilibrium data for a number of compositions of interest and for temperatures up to 400 C (750 F). Because the present study was to cover temperatures up to 700 C (1290 F), it was necessary to determine equilibria for the temperature range of 400 to 700 C (750 to 1290 F) not covered by the literature. In addition, data were taken at 300 to 400 C (570 to 750 F) for comparison with the work of Kofstad et al.

Also, some thermodynamic functions readily obtainable from the equilibrium factors were calculated.

Material

The tantalum used in this work was obtained from the Wah Chang Corporation of Albany, Oregon. It had been electron-beam melted to an ingot, 3-1/2 inches in diameter, and cold forged and swaged to a 7/16-inch diameter. Final fabrication was carried out at Battelle and consisted of

cold swaging to rod, 1/4 and 1/8 inch in diameter, and vacuum annealing for 1 hour at 1200 C (2190 F). A length of the 1/8-inch rod was cold rolled to 0.0146-inch sheet for equilibrium experiments. The 1/4-inch-diameter rod was used in the kinetic work.

The analysis of the tantalum before final fabrication is given in Table 7.

TABLE 7. ANALYSIS OF TANTALUM

Element	PPM by Weight
Al	<20
B	<1
C	42
Cb	700
Cd	<1
Cr	<20
Cu	<40
Fe	<100
Mg	<20
Mn	<20
Mo	<20
Ni	<20
Pb	<20
Si	<100
Sn	<20
Ti	<150
V	<20
W	<300
Zn	<20
H_2	<2 (a)
O_2	<20 (a)
N_2	3

Note: Average BHN 80.1.
(a) Vacuum-fusion analysis at Battelle after final fabrication.

Pure hydrogen was obtained from the thermal decomposition of uranium hydride prepared from dry tank hydrogen and degreased uranium chips.

EQUILIBRIUM STUDIES

Experimental Procedure

The method to obtain equilibrium data has been described.[9] Briefly, the data were obtained as follows: A measured quantity of hydrogen was added to the calibrated reaction tube containing approximately 4 grams of sheet tantalum. At the desired temperature, the system was allowed to come to constant (equilibrium) pressure as measured on a mercury manometer. The equilibrium composition was calculated from the equilibrium pressure, specimen weight, volume of gas addition, and gas capacity of the reaction system at the experimental room and furnace temperatures.

Results

The pressure-temperature-composition equilibria of the tantalum-hydrogen system were determined in the range 300 to 700 C (570 to 1290 F), 10 to 1000 mm Hg pressure, and atomic ratios, H/Ta, of 0. 05 to 0. 50. These atomic ratios correspond to atomic fractions, N_H, for hydrogen of about 0. 05 to 0. 333. Figure 21 is a logarithmic plot of the equilibrium pressure against the H/Ta ratio. The plot shows that the isotherms are linear at temperatures of 500 C (950 F) and higher, at lower temperatures, the isotherms show curvature which becomes increasingly marked with decrease in temperature. None of the isotherms shows an invarient pressure. This indicates that there is no two-phase region in our experimental range. It appears that such a region would appear only at some temperature below 100 C (210 F).

It had been proposed to determine equilibria at H/Ta ratios of about 0. 05, 0. 10, 0. 5, and 0. 7 and temperatures of 300 to 700 C (570 to 1290 F). Pressure was not to exceed 1000 mm of mercury. However, it was found that a 1000-mm equilibrium pressure was reached at an H/Ta ratio of 0. 5 and a temperature of only 400 C (750 F). Therefore, it was decided to eliminate the 0. 7 composition and substitute the H/Ta ratio of 0. 33 so that all the equilibrium pressures would lie below 1000 mm of mercury. The experiments were designed so that equilibrium pressures were measured for at least three different temperatures for each composition.

The equilibria were interpreted in terms of the conditions needed to form various products (solid solutions) having definite H/Ta and N_H ratios. This placed the data in the most convenient form for use in subsequent kinetic experiments. Several representative constant composition lines or isopleths are shown in Figure 22. The equilibria for a given composition

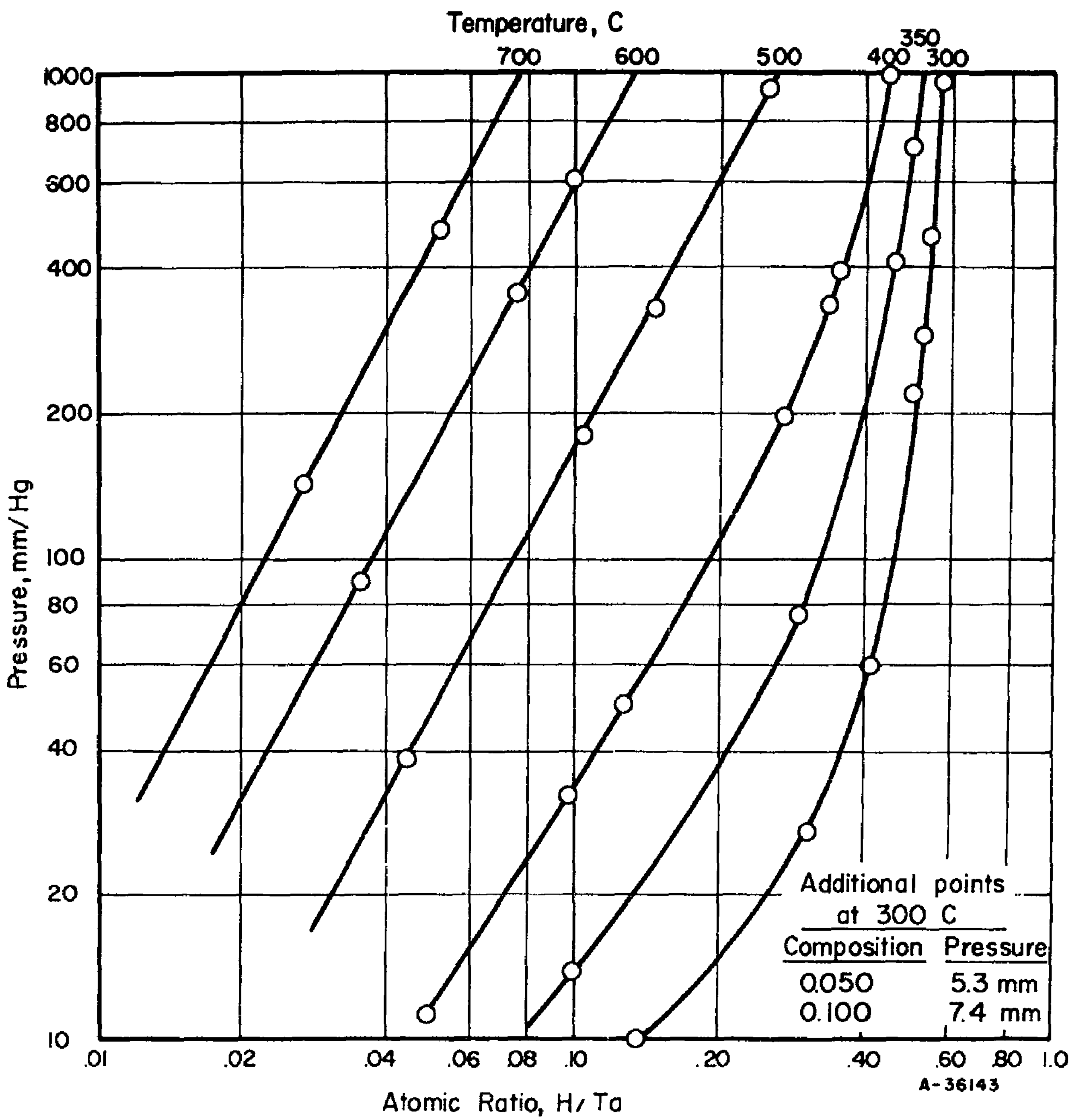


FIGURE 21. LOGARITHMIC PLOT OF ISOTHERMS IN THE Ta-H SYSTEM

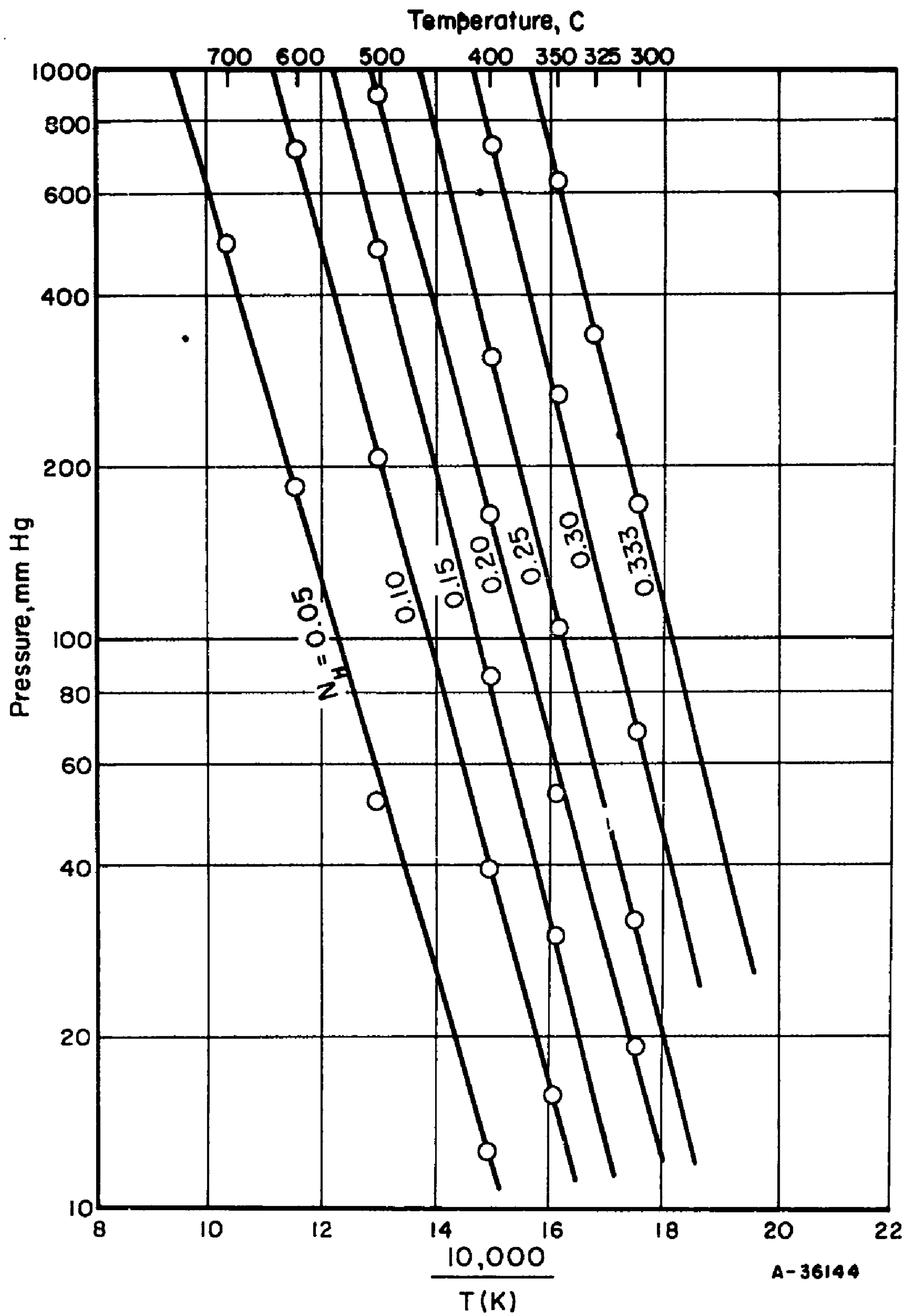


FIGURE 22. REPRESENTATIVE ISOPLETHS FOR THE Ta-H SYSTEM

in these plots of logarithm of equilibrium pressure against reciprocal temperature can be expressed as

$$\log_{10} P_{mm} = -[A/T\ (K)] + B \quad . \tag{1}$$

Values for the constants A and B determined by the method of least squares are given in Table 8.

TABLE 8. EQUATIONS FOR EQUILIBRIUM PRESSURES

N_H[(a)]	Atom Ratio, H/Ta	Constants in $\log_{10} P_{mm} = -A/T + B$	
		A	B
0.05	0.0526	3482	6.257
0.10	0.1111	3617	6.997
0.15	0.1765	3865	7.686
0.20	0.2500	3737	7.779
0.25	0.3333	3799	8.136
0.30	0.4286	3962	8.769
0.333	0.5000	4023	9.263

(a) N_H = atomic fraction of H.

Since it may be desirable to relate thermodynamic and kinetic properties rather directly, the compositions are listed primarily according to round N_H values or atomic fractions of H.

A comparison of the equilibrium pressure isotherms of the present study and those of Kofstad et al. is given in Table 9. The agreement between the two sets of data is reasonable. A point worthy of note is that the pressures for the higher concentrations are consistently lower for the present study. This may indicate that our tantalum contained less interstitial contamination, particularly oxygen, than the metal of Kofstad. As will be seen later, the lower pressures also are reflected in lower relative partial free energy values at 350 C (660 F).

Also, it appears from the collective data that the Sieverts law is obeyed approximately for the N_H composition range 0.05 to 0.20. This is evidenced by the (relative) invariance of the value of $\log \frac{\sqrt{P}}{N_H}$ for different compositions at a given temperature. Large deviations from the Sieverts law are seen for higher compositions. That these deviations are related to entropy effects rather than enthalpy changes will be shown later. Deviations were also found by Kofstad at compositions lower than those of this study.

TABLE 9. EQUILIBRIUM PRESSURE ISOTHERMS FOR Ta-H SOLID SOLUTIONS

$$\mathrm{Log} \frac{\sqrt{P_{mm}}}{N_H}$$

N_H	Temperature, C										
	300	304[a]	325	324[a]	350	349.5[a]	400	402[a]	500	600	700
0.05	1.39129	1.305	--	1.420	1.63508	1.579	1.34261	1.819	2.17702	2.43489	2.64008
0.10	1.34262	1.305	--	1.420	1.59550	1.579	1.81151	1.819	2.16256	2.42700	--
0.15	1.29447	1.305	--	1.420	1.56490	1.579	1.79541	1.819	2.16685	--	--
0.20	1.32746	1.307	--	1.423	1.58950	1.581	1.81191	1.822	2.17143	--	--
0.25	1.35504	1.351	--	1.470	1.62118	1.637	1.84757	1.882	--	--	--
0.30	1.45040	1.487	--	1.613	1.72754	1.757	1.96395	(2.011)[b]	--	--	--
0.333	1.59861	1.649	1.74567	1.777	1.88008	(1.909)[b]	2.12404	(2.158)[b]	--	--	--

(a) Data from Reference (4).

(b) Values extrapolated from low-temperature isotherms.

THERMODYNAMIC FUNCTIONS

When the dissolution of hydrogen in tantalum is expressed as

$$1/2\,H_2\,(\text{gas}) \rightleftharpoons H_i\,(\text{dissolved in Ta}), \tag{2}$$

the relative partial free energy of hydrogen in a given Ta-H solution (i) is

$$\bar{F}_{H_i} - 1/2 F^{\circ}_{H_2} = RT \ln P_{H_2}^{1/2} \ . \tag{3}$$

P_{H_2} is the pressure of hydrogen gas in equilibrium with the solution at temperature, T, and R is the gas constant.

The values for the partial molar enthalpies, $\bar{H}_H - \frac{H^{\circ}_{H_2}}{2}$, and entropies, $\bar{S}_H - \frac{S^{\circ}_{H_2}}{2}$, were calculated by the method of least squares from plots of $\log\sqrt{P}$ against reciprocal temperature. This treatment also yielded the probable errors for these values. The free energy values were then calculated according to the Gibbs-Helmholtz equation

$$\Delta F = \Delta H - T\Delta S \quad , \tag{4}$$

which applies to both the integral and partial molar quantities. The calculated partial molar energy values for hydrogen in solution in tantalum are given in Table 10.

The partial molar enthalpy (the negative of the heat of vaporization) decreases systematically with increasing hydrogen content, if the value for $N_H = 0.15$ is omitted. This indicates an increase in hydrogen binding in the Ta-H lattice over the N_H range 0.05 to 0.333. This agrees with the conclusions of Kofstad who reported an increase in partial heat of vaporization.

The partial molar entropy also decreases rapidly with increasing hydrogen contents. This is because of the decreasing availability of interstitial sites as more hydrogen is added to the metal. Thus, the enthalpy and entropy changes in the tantalum-hydrogen system make for opposite deviations in behavior of the Sieverts law. However, the entropy effect is much stronger so that abnormal increases in pressure are observed with increasing hydrogen concentrations. This, of course, is a demonstration of a rise in partial free energy, $\bar{F} - \frac{F^{\circ}_{H_2}}{2}$, such as shown by calculated values for 350 C (660 F) given in Table 10.

TABLE 10. PARTIAL MOLAR THERMODYNAMIC FUNCTIONS(a) FOR HYDROGEN IN Ta-H SOLID SOLUTIONS

N_H:	0.05	0.10	0.15	0.20	0.25	0.30	0.333
			This Study				
Temperature Range, C	400 to 700	350 to 600	300 to 500	300 to 500	300 to 400	300 to 400	300 to 350
$\bar{F}_H - \frac{F^\circ_{H_2}}{2}$ cal/g-atom(b)	-3149	-2406	-1987	-1566	-1202	-673	-104
$\bar{S}_H - \frac{S^\circ_{H_2}}{2}$ eu/g-atom	-7.73 ± 0.14(c)	-9.42 ± 0.10	-11.00 ± 0.04	-11.21 ± 0.29	-12.02 ± 0.21	-13.47 ± 0.40	-14.60 ± 0.03
$\bar{H}_H - \frac{H^\circ_{H_2}}{2}$ cal/g-atom	-7965 ± 115(c)	-8275 ± 75	-8840 ± 25	-8550 ± 190	-8690 ± 130	-9065 ± 245	-9200 ± 15
			From Reference (4) for 350 C				
$\bar{F} - \frac{F^\circ_{H_2}}{2}$ cal/g-atom	-3310	-2510	-2010	-1600	-1210	-520	-1
$\bar{S}_H - \frac{S^\circ_{H_2}}{2}$ eu/g-atom	-9.9	-11.1	-12.1	-12.7	-13.6	-14.8	-14.9
$\bar{H}_H - \frac{H^\circ_{H_2}}{2}$ cal/g-atom	-9500	-9400	-9500	-9500	-9700	-9700	-9300

(a) The relative partial molar free energies, enthalpies, and entropies are for 1 g-atom of hydrogen.
(b) These free-energy values are for 350 C only.
(c) The ± values in this table are probable error.

The partial molar enthalpies and entropies of the tantalum-hydrogen system show the same trends and are of the same general magnitude as those of the columbium-hydrogen system.[10] However, for a given temperature and composition, the tantalum-hydrogen system shows the higher dissociation pressure of hydrogen, i. e., the partial molar free energy for the dissolution reaction is higher.

An equation for calculating the partial molar free energy for tantalum in a given solution and the total free energy of formation of the solution was obtained by modification of the Duhem-Margules equation,[11]

$$N_{Ta}\, d\,\overline{F}_{Ta} + N_H\, d\,\overline{F}_H = 0 \quad . \tag{5}$$

N is the mole or atom fraction and $N_{Ta} + N_H = 1$. By differentiation we obtain $d\,N_{Ta} + d\,N_H = 0$. Multiplying the first term by $\frac{N_{Ta}}{N_{Ta}}$ and the second by $\frac{N_H}{N_H}$ (i. e., unity),

$$N_{Ta} \frac{d\,N_{Ta}}{N_{Ta}} + N_H \frac{d\,N_H}{N_H} = 0$$

or

$$N_{Ta}\, d \ln N_{Ta} + N_H\, d \ln N_H = 0 \quad . \tag{6}$$

From Equation (3)

$$d\,\overline{F}_H = RT\, d \ln P_{H_2}^{1/2} \tag{7}$$

at constant temperature. Transposing one term of Equation (5) dividing by N_{Ta} and then substituting the equivalent of $d\,\overline{F}_H$ from Equation (7)

$$d\,\overline{F}_{Ta} = -\frac{N_H}{N_{Ta}}\, d\,\overline{F}_H = -RT \frac{N_H}{N_{Ta}}\, d \ln P_{H_2}^{1/2} \quad . \tag{8}$$

Also, from Equation (6) by multiplying by RT and dividing by N_{Ta}

$$RT\, d \ln N_{Ta} + RT \frac{N_H}{N_{Ta}} \ln N_H = 0 \quad . \tag{9}$$

Subtracting Equation (9) from (8)

$$d\,\overline{F}_{Ta} = RT\, d \ln N_{Ta} - RT \frac{N_H}{N_{Ta}}\, d \ln \frac{P_{H_2}^{1/2}}{N_H} \quad . \tag{10}$$

Integrating, we obtain

$$\overline{F}_{Ta_i} - F^{\circ}_{Ta} = RT \left[\ln N_{Ta_i} - \int_{N_H = 0}^{N_{H_i}} \frac{N_H}{N_{Ta}} \, d \ln \frac{P^{1/2}_{H_2}}{N_H} \right] , \qquad (11)$$

which is the equation for the partial molar free energy for tantalum in solution. The standard reference states at any temperature are 1 atmosphere pressure for hydrogen and the pure hydrogen-free condition for tantalum.

The negative integral in Equation (11) is the ln of the activity coefficient of tantalum in a solution (i). To solve the equation it is necessary to graphically integrate the last term. To help visualize the areas to be integrated it is useful to consider the case of integration by parts where the negative integral becomes

$$-N_{H_i} / N_{Ta_i} \ln \frac{P^{1/2}_{H_2}}{N_{H_i}} + \int_{N_H = 0}^{N_{H_i}} \ln \frac{P^{1/2}_{H_2}}{N_H} \, d\, N_{H_i}/N_{Ta_i}$$

Figure 23 shows several equilibrium isotherms plotted for integration. The 500 and 600 C (930 and 1110 F) curves approach the horizontal position which would indicate obedience of Sieverts' law. Figure 24 is a plot of a typical curve expanded for graphical integration. The negative and positive areas involved for the composition, $N_H = 0.25$ are indicated. The regions of overlap where the terms cancel each other are shown in crosshatch. The computed partial molar free energies for tantalum are listed in Table 11.

The equation for the total free energy of formation for a given solution (i) is a combination of Equations (3) and (6).

$$\Delta F_{f_i} = N_{H_i} (\overline{F}_{H_i} - \tfrac{1}{2} F^{\circ}_{H_2}) + N_{Ta_i} (\overline{F}_{Ta_i} - F^{\circ}_{Ta}) \quad . \qquad (12)$$

Values for ΔF_{f_i} are given in Table 12. Equations of the same basic form as Equations (11) and (12) were used by Katz and Gulbransen(10) for calculating the thermodynamic functions for the columbium-hydrogen system.

Both the partial molar free energy values (for tantalum) versus temperature plots and the total free energy versus temperature plots for each composition were linear. Therefore, the respective enthalpy and entropy functions and their probable errors could be calculated by a least squares treatment of the data for a given composition. It follows that both functions were invariant over the experimental temperature range. The partial molar enthalpies and entropies of tantalum in several hydrogen-in-tantalum solutions are given in Table 13. Corresponding total enthalpies and entropies for these solutions ranging in compositions from $N_H = 0.05$ to 0.333 are given in Table 14.

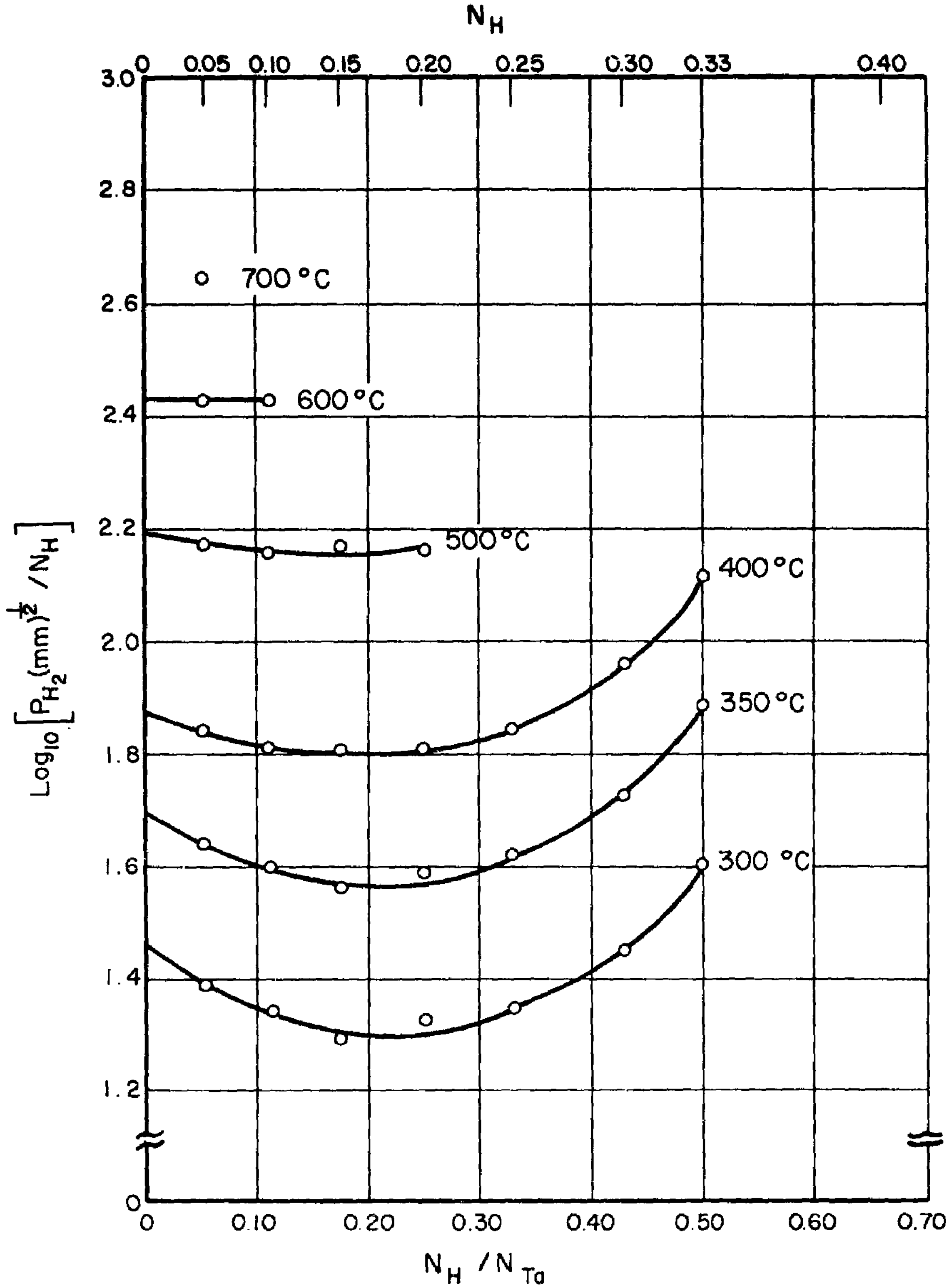


FIGURE 23. EQUILIBRIUM ISOTHERMS FOR THE TANTALUM-HYDROGEN SYSTEM

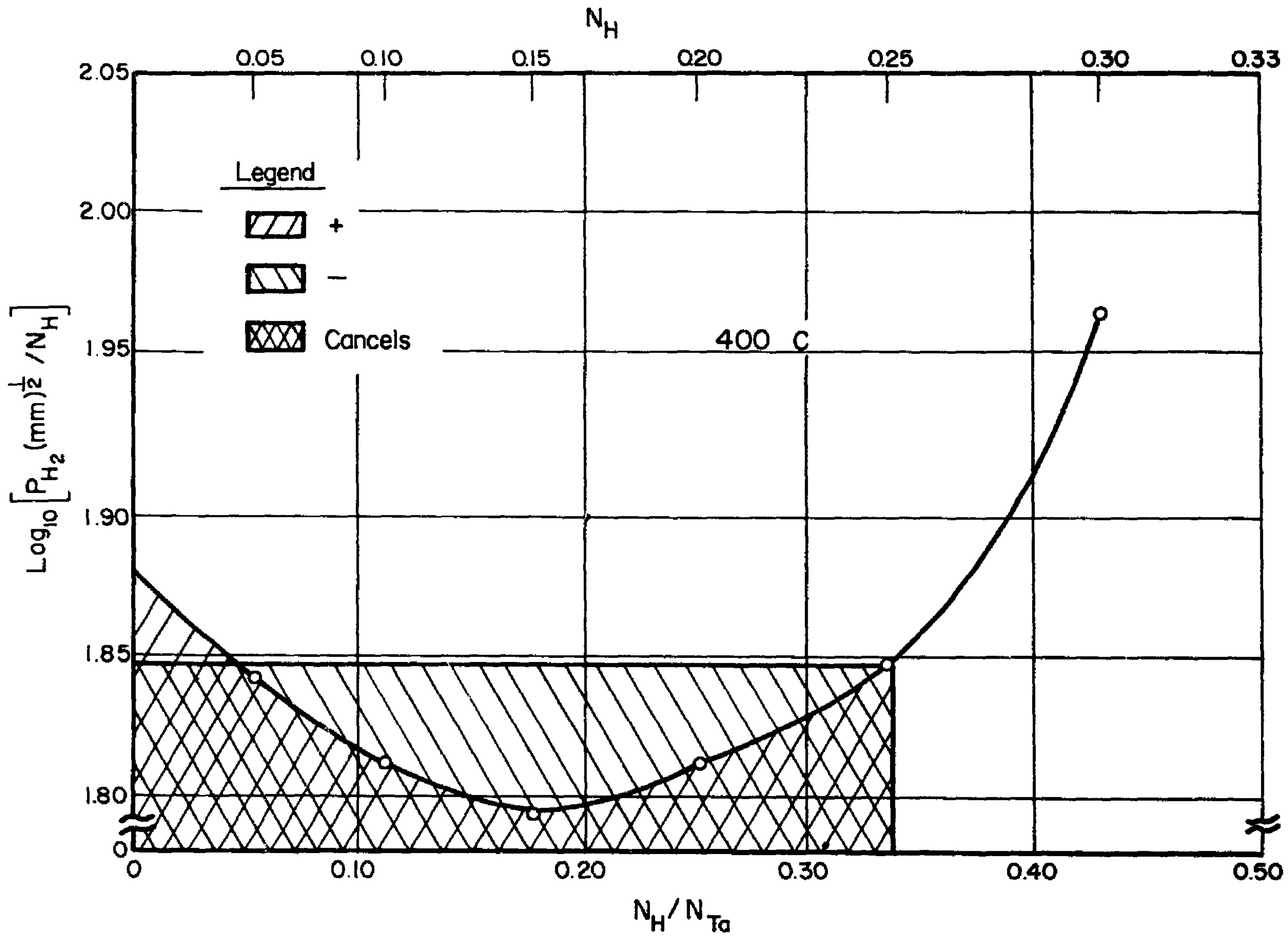


FIGURE 24. INTEGRATION FOR $N_H = 0.25$ AT 400 C

TABLE 11. PARTIAL MOLAR-FREE ENERGIES FOR TANTALUM IN TANTALUM-HYDROGEN SOLUTIONS

N_{Ta}	$F_{Ta_i} - F^{\circ}_{Ta}$ (Cal/Mole of Alloy)					
	Temperature, C					
	300	350	400	500	600	700
0.95	-54.0	-56.8	-62.8	-77.0	-87.4	-99.2
0.90	-105	-113	-128	-156	-179	--
0.85	-159	-175	-198	-246	--	--
0.80	-222	-257	-286	-341	--	--
0.75	-329	-374	-408	--	--	--
0.70	-527	-576	-640	--	--	--
0.666	-754	-839	-935	--	--	--

TABLE 12. TOTAL FREE ENERGIES OF FORMATION IN THE TANTALUM-HYDROGEN SYSTEM

N_H	ΔF_f (Cal/Mole of Alloy)						
	Temperature, C						
	300	350	350[(a)]	400	500	600	700
0.05	-228	-211	(-230)	-198	-173	-144	-116
0.10	-382	-343	(-370)	-308	-240	-166	--
0.15	-516	-447	(-470)	-348	-260	--	--
0.20	-603	-518	(-540)	-430	-250	--	--
0.25	-697	-581	(-600)	-457	--	--	--
0.30	-773	-605	(-620)	-448	--	--	--
0.333	-780	-593	(-600)	-414	--	--	--

(a) Data from Reference (8).

TABLE 13. PARTIAL MOLAR ENTHALPIES[a] AND ENTROPIES[b] OF TANTALUM IN TANTALUM-HYDROGEN SOLUTIONS

N_{Ta}	$\bar{H}_{Ta_i} - H^{\circ}_{Ta}$	Probable Error	$\bar{S}_{Ta_i} - S^{\circ}_{Ta}$	Probable Error
0.95	14.8	±2.1	0.117	±0.003
0.90	42.9	±4.1	0.255	±0.006
0.85	100	±11	0.445	±0.017
0.80	115	±10	0.593	±0.015
0.75	127	±27	0.798	±0.043
0.70	119	±37	1.12	±0.06
0.666	286	±26	1.81	±0.04

(a) Cal/mole of alloy.
(b) Cal/deg-mole of alloy.

TABLE 14. TOTAL ENTHALPIES[a] AND ENTROPIES[b] OF FORMATION IN THE TANTALUM-HYDROGEN SYSTEM

					Kofstad's Data	
N_H	ΔH_f	Probable Error	ΔS_f	Probable Error	ΔH_f	ΔS_f
0.05	-384	±2	-0.275	±0.003	-480	-0.40
0.10	-789	±4	-0.713	±0.005	-940	-0.92
0.15	-1242	±10	-1.27	±0.01	-1410	-1.5
0.20	-1618	±8	-1.76	±0.01	-1860	-2.1
0.25	-2077	±20	-2.41	±0.03	-2370	-2.8
0.30	-2636	±27	-3.25	±0.04	-2860	-3.6
0.333	-2873	±17	-3.66	±0.03	-3180	-4.08

(a) Cal/mole of alloy.
(b) Cal/deg-mole of alloy.

Data of Kofstad[8] et al. are listed for comparison in Tables 12 and 14. Although their total enthalpies ranged from 96 to 307 calories per mole higher than those of our study their total free energies were higher by only 7 to 27 calories per mole.

The uncertainties in the values of $\overline{H}_{Ta_i} - H^\circ_{Ta}$ and $\overline{S}_{Ta_i} - S^\circ_{Ta}$ in the present study are from 9 to 30 per cent and 2 to 5 per cent, respectively. Since the investigations of similar systems have either omitted presenting uncertainties in values for these functions or even the values themselves, no comparison can be made. However, because of the indirect means of deriving these values the uncertainties appear quite reasonable. It is seen that the uncertainties in the values for partial molar energy functions for tantalum have an insignificant effect on the total energy functions. This is evidenced by the probable errors for ΔH_f and ΔS_f which are of the order of only 1 per cent.

KINETIC STUDIES

Kinetic data for the sorption of hydrogen by tantalum to produce compositions having N_H values of 0.05, 0.10, 0.25, and 0.33 were obtained in the temperature range of 300 to 700 C (570 to 1290 F). Experimental conditions of temperature and pressure were determined from the isopleth of the desired product as determined in the equilibrium studies.

Samples for kinetic studies were made from the same stock used for the equilibrium studies. They consisted of cylinders 0.6 to 0.7 cm in diameter and 2.5 to 3.5 cm long. Swaged rods were filed clean and finish dry-abraded through 240, 400, and 600 grit silicon carbide papers. Samples were spot welded to the bead of a platinum-platinum plus 10 per cent rhodium thermocouple and suspended by the thermocouple in a Vycor reaction tube of a modified Sieverts apparatus.[12] The system was evacuated to a pressure of less than 0.01 micron of mercury. The sample was then activated by heating with a resistance-wound furnace at 900 C (1650 F) for 1 hour before adjusting the temperature to that of the run. This treatment served to remove dissolved hydrogen and dissolve surface oxide films thus minimizing incubation periods for reaction. Although eleven samples were used in the kinetic studies, four of them were reused 3 to 11 times without removal from the reaction tube. Reacted hydrogen was removed between runs by the 900 C (1650 F) heating in vacuum. This treatment had no apparent effect on the physical properties of the metal.

The reaction was initiated by admitting hydrogen through the pressure regulator to the reaction tube. The buret was kept balanced at atmospheric pressure at all times. Readings of the buret were taken at convenient time intervals depending on the speed of the reaction. The amount of gas reacted

with the sample was the difference between the volume added from the buret and the volume remaining in the gas phase in the calibrated dead space of the reaction tube.

Representative rate data are shown in Figures 25 through 27. The parabolic character of the raw data with some initial deviation is apparent for reactions at and above 500 C (930 F) but becomes obscure at lower temperatures. Since no compound was formed, the reaction was expected to be one of simple dissolution and diffusion of hydrogen in tantalum accompanied by possible secondary interface reactions. Accordingly interpretation of the data was attempted in terms of theoretical diffusion behavior.

Fick's second law for diffusion in a finite cylinder may be expressed as[9]

$$\frac{C}{C_o} = 1 - \frac{32}{\pi^2} \sum_{n=0}^{\infty} \sum_{m=1}^{\infty} \frac{1}{\beta_m^2 \; (2n+1)^2} \exp\left\{\left[-\frac{Dt}{a^2} \; \frac{(2n+1)^2 \pi^2}{\left(\frac{\ell}{a}\right)^2} + \beta_m^2\right]\right\} \quad , \qquad (13)$$

where

a = radius of sample

ℓ = length of sample

D = diffusion coefficient

C = average hydrogen concentration

t = time

C_o = the final equilibrium concentration

β_m = the m^{th} root of the Bessel function J_o (β) and the indices m and n range through integral values as desired.

Since the length of radius ratio, $\frac{\ell}{a}$, of the experimental cylindrical samples varied from about 5 to 12, theoretical curves were calculated from Equation (13) to permit analysis of data for the sorption of gas by any cylinder. These curves are shown in Figure 28 where C/C_o is plotted against $\frac{\sqrt{Dt}}{a}$ for $\frac{\ell}{a}$ ratios of 1, 2, 4, 8, 16, and infinity.

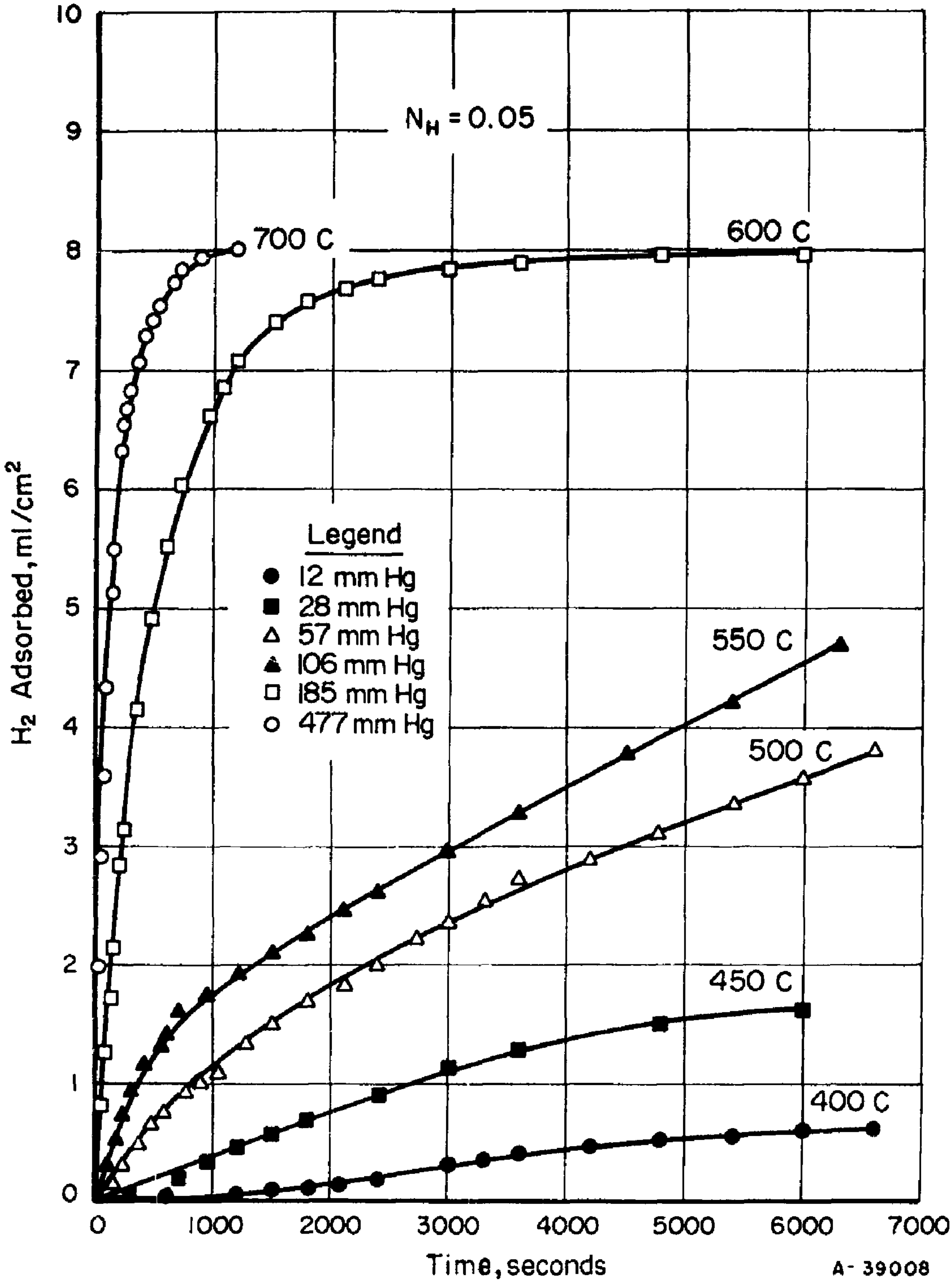


FIGURE 25. REPRESENTATIVE RATE DATA FOR THE REACTION OF HYDROGEN WITH TANTALUM (N_H = 0.05)

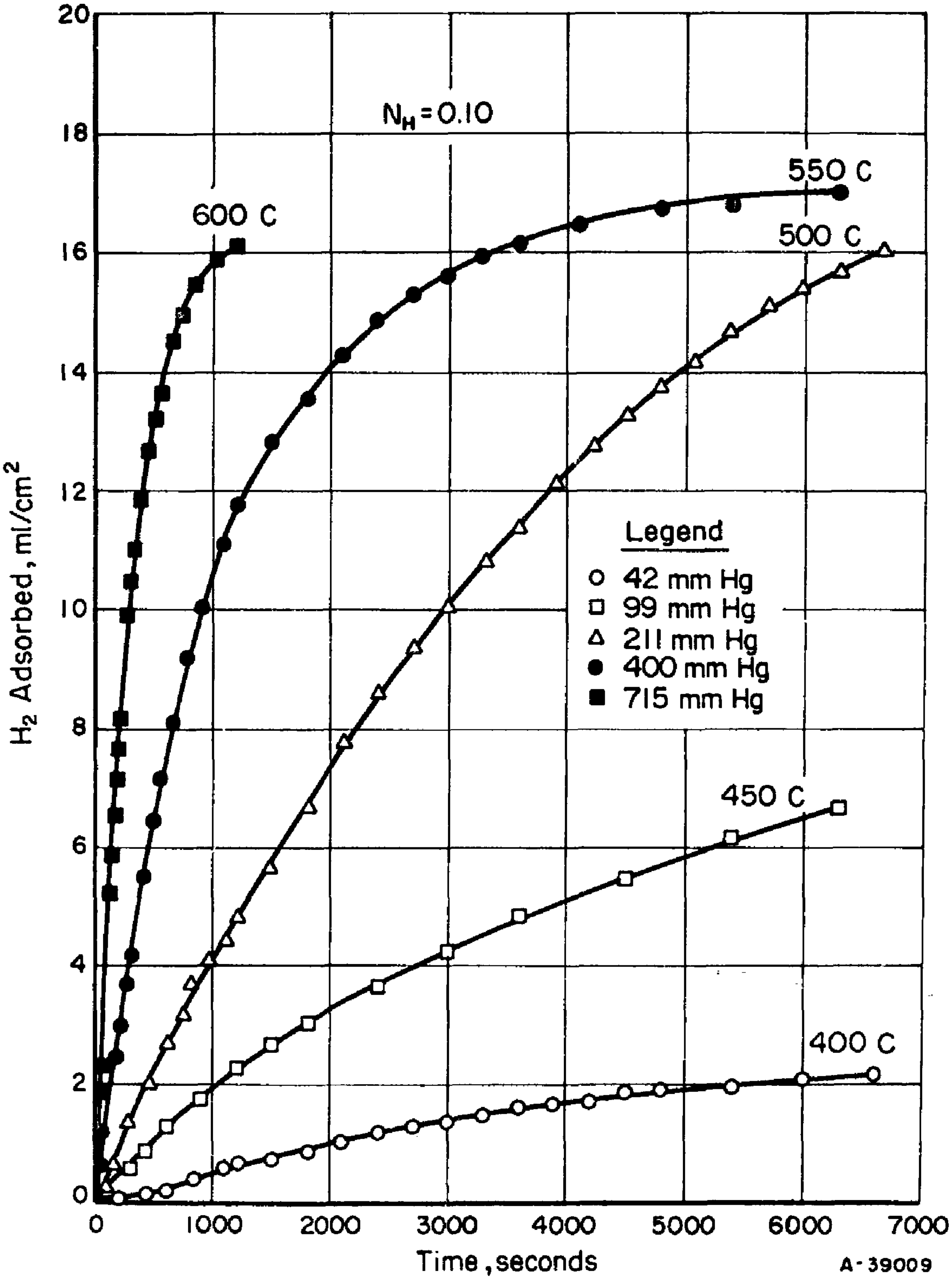


FIGURE 26. REPRESENTATIVE RATE DATA FOR THE REACTION OF HYDROGEN WITH TANTALUM (N_H = 0. 10)

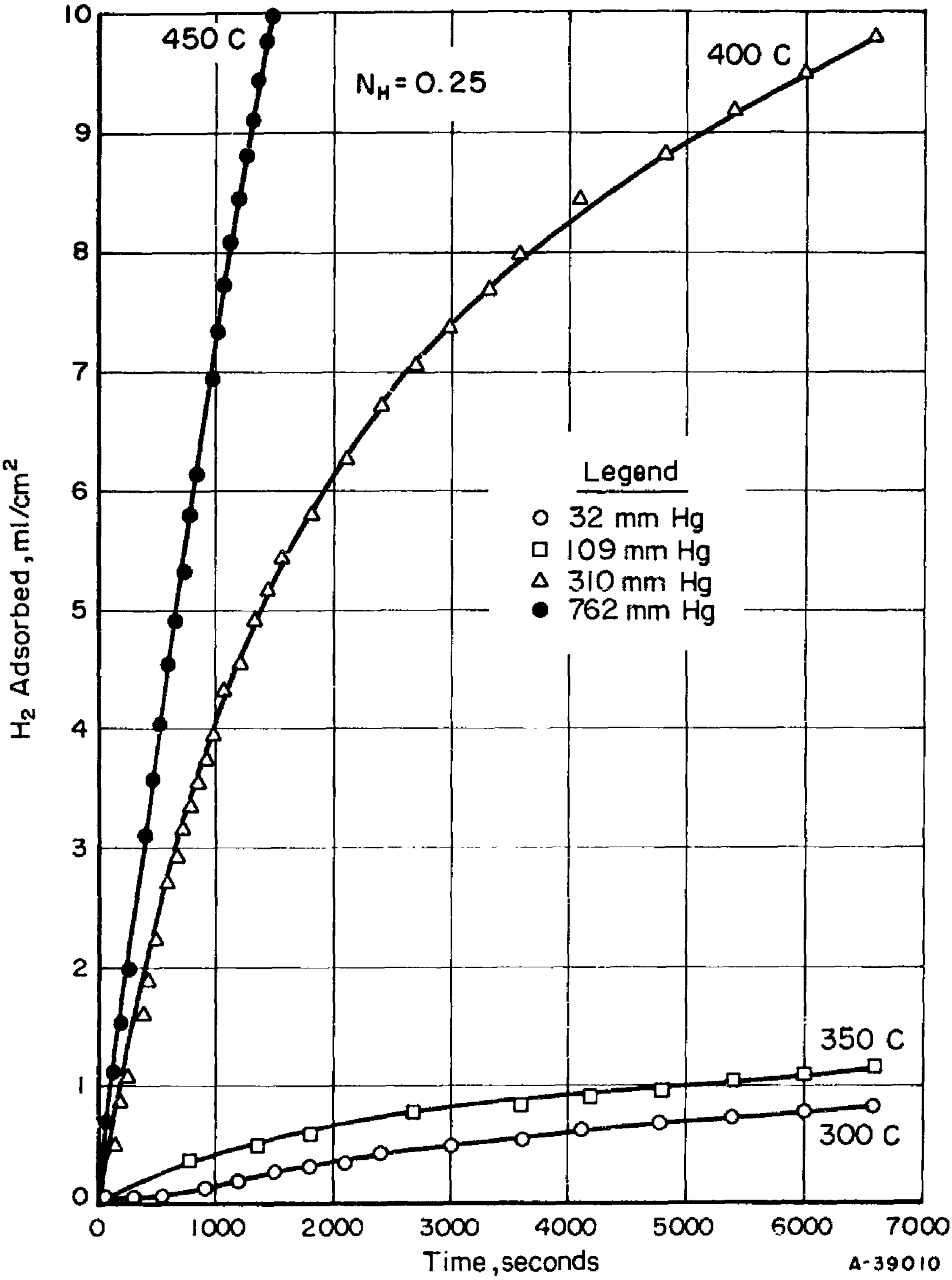


FIGURE 27. REPRESENTATIVE RATE DATA FOR THE REACTION OF HYDROGEN WITH TANTALUM (N_H = 0.25)

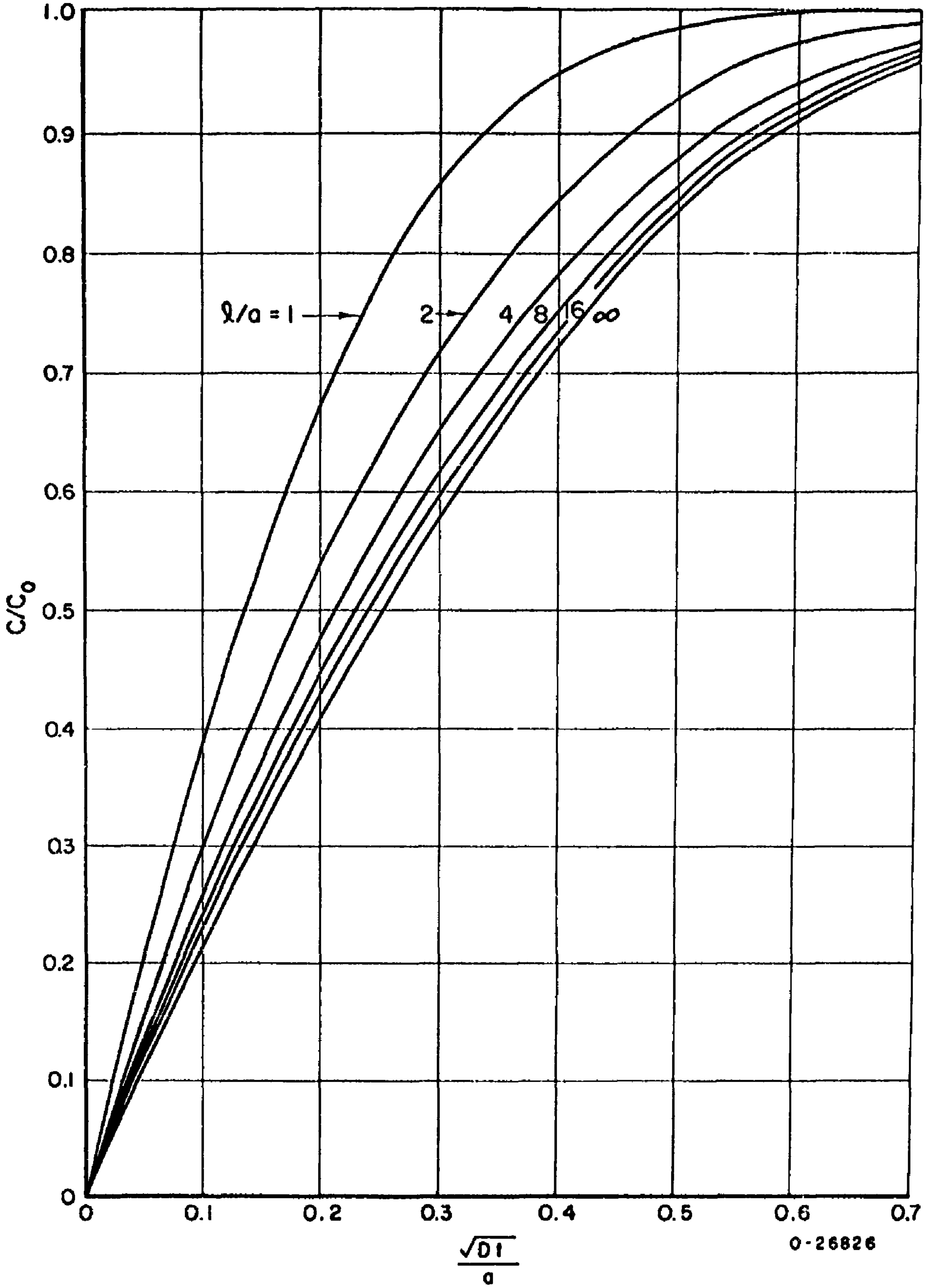


FIGURE 28. THEORETICAL CURVES FOR SORPTION IN CYLINDERS

The fitting of the experimental data to the theoretical diffusion curves of Figure 28 becomes impractical if not impossible when the experimental reaction involves induction periods. However, Demarez, Hoch, and Meunier[14] offer a solution to this problem. They have shown that for values of t greater than some threshold time, t_o, the series such as appears in Equation (13) degenerate to their first terms with adequate accuracy for interpretation of the experimental data. Then

$$\frac{C}{C_o} = 1 - \frac{32}{\pi^2 \beta_1^2} \exp - \frac{Dt}{a^2}\left[\frac{\pi^2}{\left(\frac{\ell}{a}\right)^2} + \beta_1^2\right] \quad . \tag{14}$$

Converting to logarithmic form one obtains:

$$\log (1 - C/C_o) = \log \frac{32}{\pi^2 \beta_1^2} - \frac{Dt}{2.303a^2}\left[\frac{\pi^2}{(\ell/a)^2} + \beta_1^2\right] \quad . \tag{15}$$

Since β_1, ℓ, and a are known, D can be determined from the slope of the straight line plot of $\log (1-C/C_o) = f(t)$. That is, slope = -(const.) D/a^2. Re-examination of the values from the double series from which the curves of Figure 28 were drawn revealed that they also could be plotted similarly as a function of t to yield lines which were adequately straight over reasonably large ranges. The intercepts of the lines differed from the calculated intercept and decreased with increasing values of ℓ/a. The slopes differed only 2 or 3 per cent from those calculated from Equation (15). The more refined values of slopes and intercepts calculated from the data in Figure 8 were used in our treatment of the experimental data.

Figure 29 shows representative plots of $\log 1 - C/C_o$ versus time. At 600 C (1110 F) the reaction was quite rapid and approached complete saturation in the experimental time. At 500 C (930 F) the reaction was much slower and reached only 60 per cent saturation in 3 hours. With the D values calculated from these curves, the data were plotted as in Figure 30 for comparison with the theoretical diffusion curves. It is seen that both the 600 and 500 C (1110 and 930 F) curves show the initial "fast" reaction called for by theory and then parallel the straight line portion of the theoretical curve. The experimental curves lie above the theoretical curves because of retardation of the initial period of reaction.

Experimental diffusion coefficients for the tantalum-hydrogen systems at compositions of N_H = 0.05, 0.10, 0.25, and 0.33 over a temperature range of 300 to 700 C (570 to 1290 F) are given in Table 15. Variation of diffusion coefficient as a function of temperature is shown for N_H = 0.05 in Figure 31 and N_H = 0.10 in Figure 32. A scatter of points for N_H = 0.25 and N_H = 0.33 also appears in Figure 32. They indicate the tendency toward

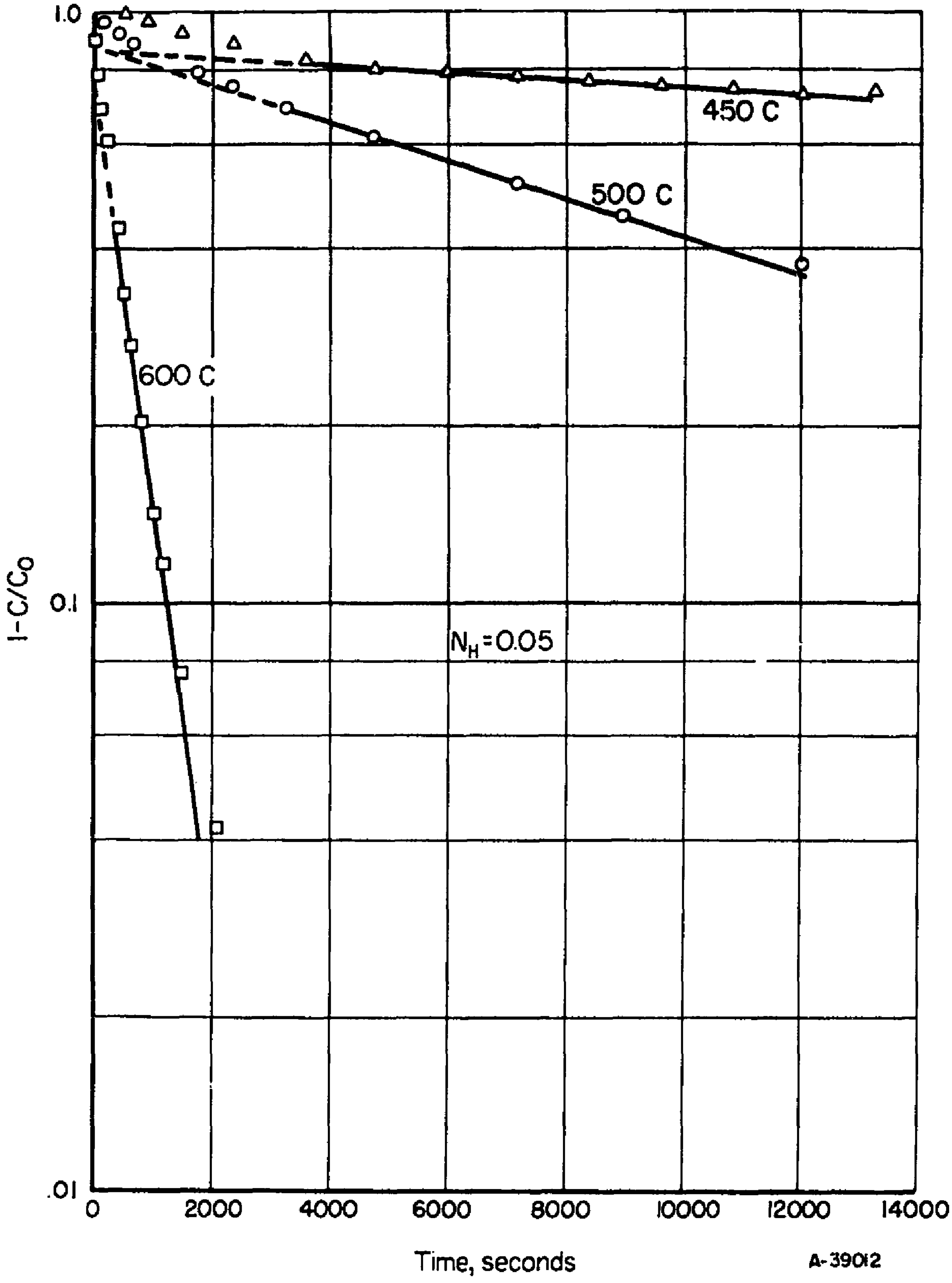


FIGURE 29. HYDROGEN ABSORPTION AS A FUNCTION OF TIME (N_H = 0.05)

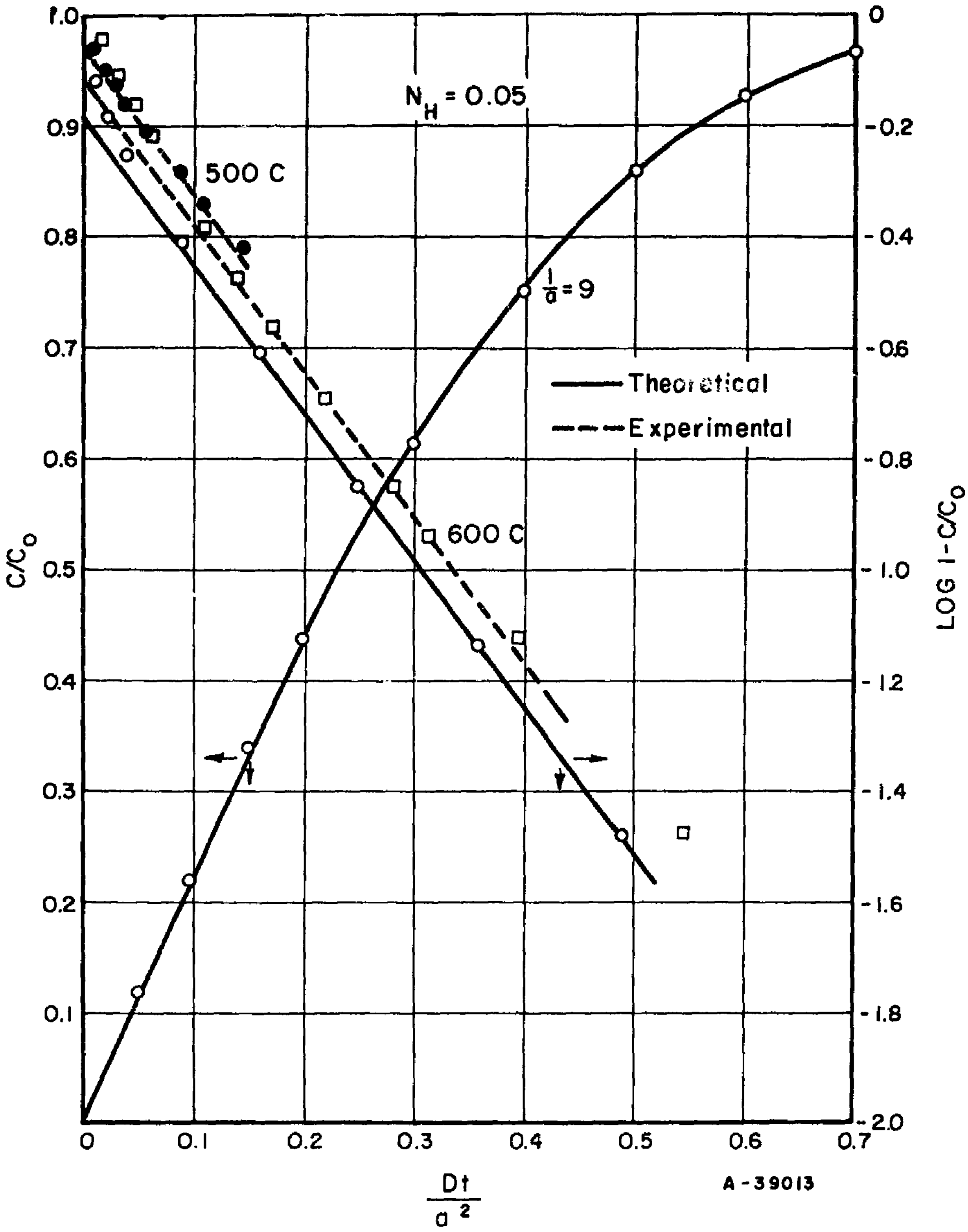


FIGURE 30. COMPARISON OF THEORETICAL AND EXPERIMENTAL ABSORPTION RATES (N_H = 0.05)

TABLE 15. DIFFUSION COEFFICIENTS FOR THE TANTALUM-HYDROGEN SYSTEM

Temperature, C	Equilibrium Pressure, mm Hg	Composition, N_H	Diffusion Coefficient, $D \times 10^6$ cm^2/sec
300	32	0.25	0.033
	175	0.33	0.356
350	109	0.25	0.038
	638	0.33	0.114
	638	0.33	0.441
400	12	0.05	0.125
	42	0.10	0.273
	310	0.25	0.614
450	28	0.05	0.239
	99	0.10	0.803
	762	0.25	1.87
500	57	0.05	1.42
	211	0.10	5.77
550	106	0.05	2.20
	400	0.10	16.0
600	185	0.05	8.42
	185	0.05	26.8
	185	0.05	30.4
	715	0.10	47.5
	715	0.10	55.4
650	306	0.05	23.8
	306	0.05	24.0
700	477	0.05	74.5
	477	0.05	85.6

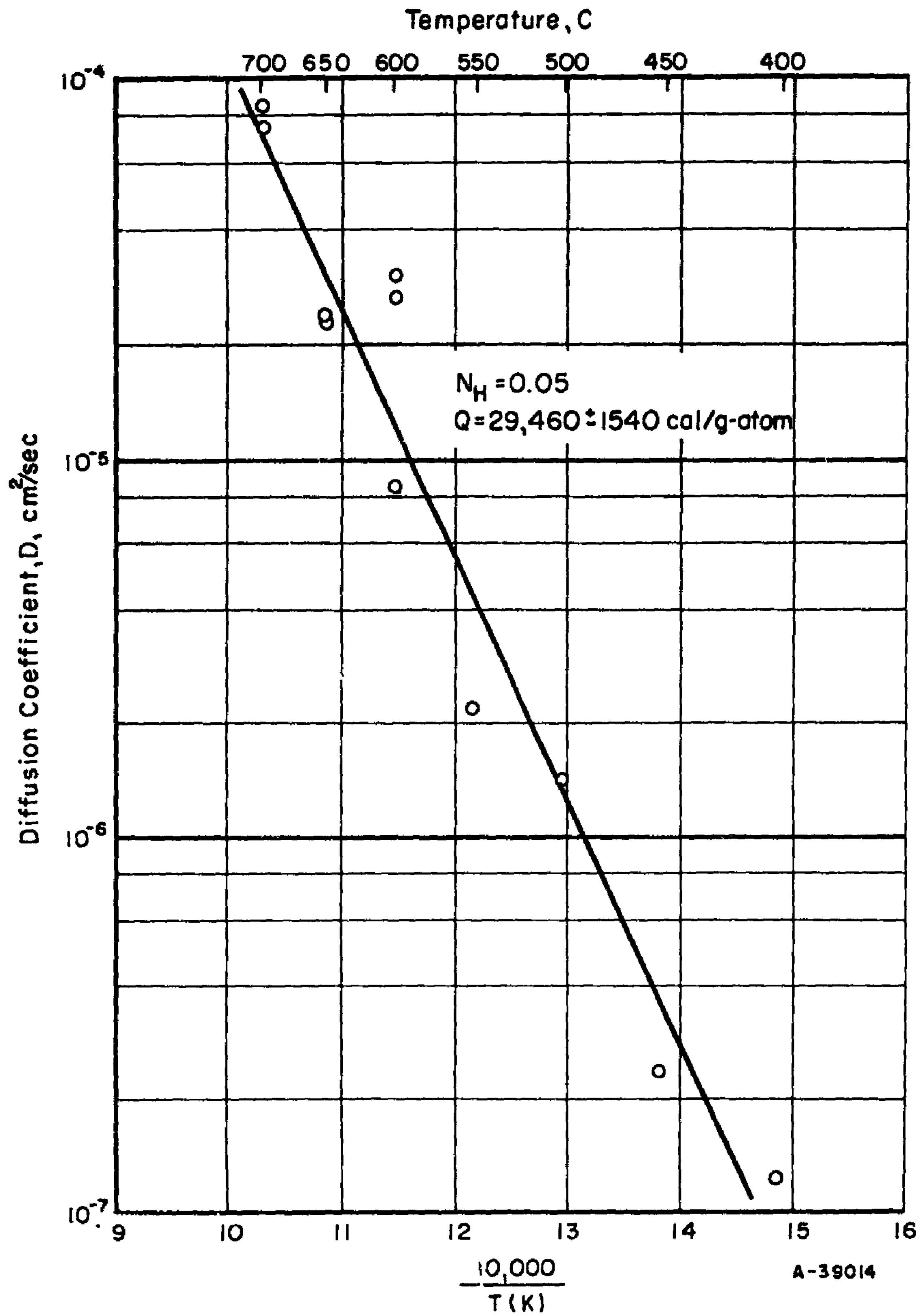


FIGURE 31. TEMPERATURE DEPENDENCE OF THE DIFFUSION COEFFICIENT FOR HYDROGEN IN TANTALUM ($N_H = 0.05$)

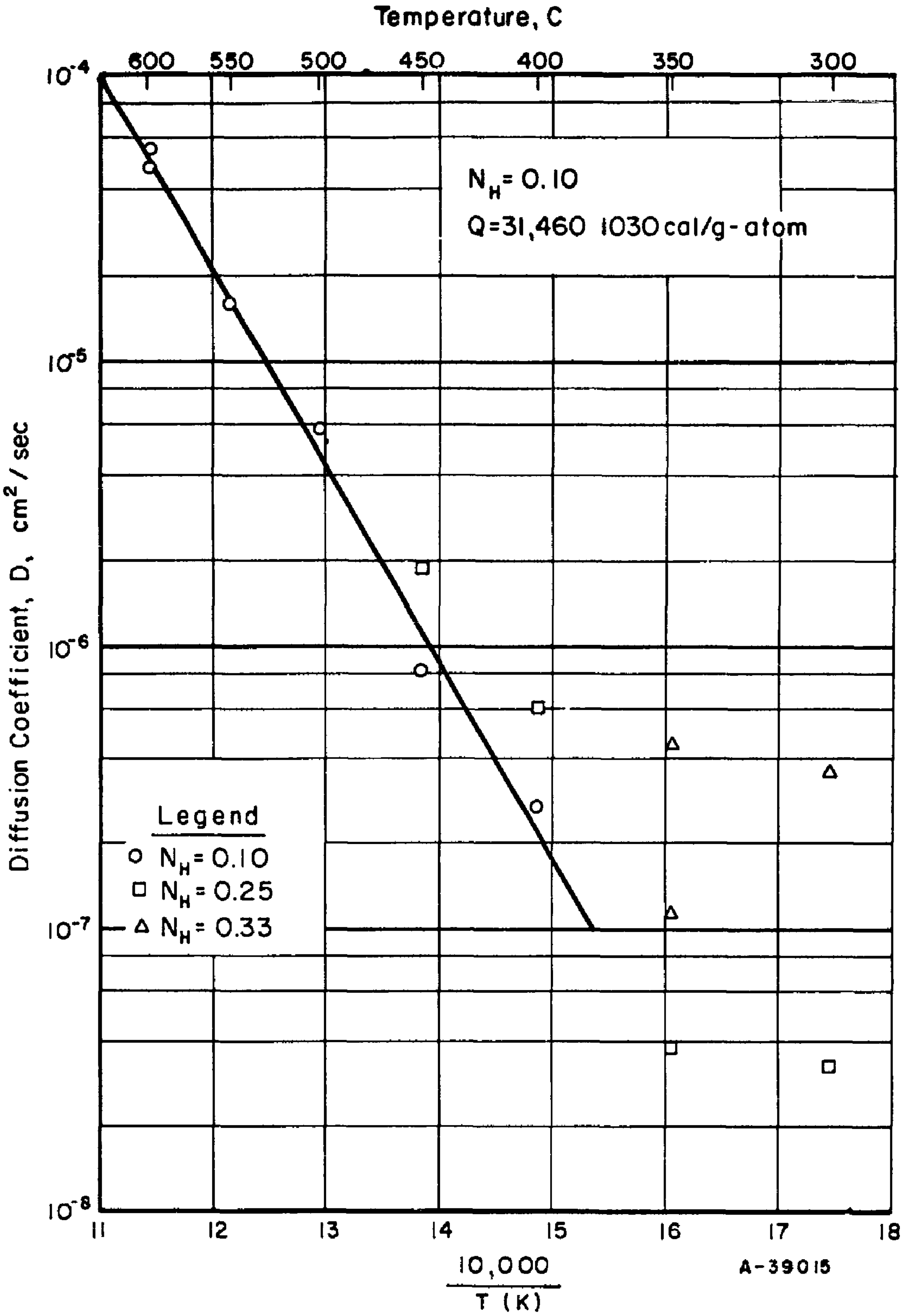


FIGURE 32. TEMPERATURE DEPENDENCE OF THE DIFFUSION COEFFICIENT FOR HYDROGEN IN TANTALUM (N_H = 0.10)

faster diffusion rates at the higher pressures required to form the solutions with larger atom fractions of hydrogen.

The equations for the best straight lines in Figures 31 and 32 were determined by the method of least squares. The equations are

$$D_{N_H=0.05} = 305.\ \exp[(-29{,}460 \pm 1540)/RT]\ \mathrm{cm}^2/\mathrm{sec} \qquad (16)$$

and

$$D_{N_H=0.10} = 3790.\ \exp[(-31{,}460 \pm 1030)/RT]\ \mathrm{cm}^2/\mathrm{sec}\ , \qquad (17)$$

where the activation energy for hydrogen diffusion is 29,460 ± 1540 cal/g-atom for composition N_H=0.05 and 31,460 ± 1030 cal/g-atom for composition N_H=0.10. It is seen that these values of activation energy agree within the limits of experimental error.

Since Equations (16) and (17) have the form $D = D_o \exp(-E/RT)$ the entropy of diffusion can be calculated from D_o and E by applying the theory of Wert and Zener[15, 16] for interstitial diffusion. For a body-centered-cubic lattice

$$D_o = 1/6\ a_o^2\ \nu \exp(\Delta S/R) \qquad (18)$$

where a_o, the lattice constant is 3.296 A for tantalum. The vibration frequency, ν, of a solute atom in an interstitial position is given by the approximation

$$\nu = (E/2m\lambda^2)^{1/2}\ , \qquad (19)$$

where E is assumed approximately equal to the activation energy, m is the mass of the solute atom, and λ, the distance between interstitial positions, is assumed to be $a_o/2$. From Equation (18), ΔS for diffusion of hydrogen in tantalum was calculated to be 20.8 cal/g-atom degree. Theory indicates that a positive ΔS is characteristic of interstitial diffusion and not diffusion through grain boundaries or other short circuiting paths.

Discussion

It was observed that the activation energies for diffusion of hydrogen in tantalum obtained in the present study are considerably higher than the few available literature values for diffusion of hydrogen in metals. Typical values in cal/g-atom are 12,380 for α titanium,[17] 6640 for β titanium,[17] 7060 for α zirconium,[18] and 9370 for columbium.[9] Also the experimental value for D_o is several orders of magnitude higher than those reported for

most other hydrogen-metal systems. The high D_0 value leads to a higher than usual value (20.8 cal/g-atom degree) of ΔS, the entropy of diffusion. However, Albrecht and Goode[19] report an equation for diffusion of hydrogen in β zirconium, $D = 6.14 \times 10^4 \exp(-45{,}900/RT)$, in which all the factors and the calculated ΔS, 30.7 cal/g-atom degree, are of the same magnitude as those of the tantalum study. This is of considerable interest since the diffusion coefficient for zirconium was obtained by a different method, that of permeation. Further data on the diffusion of hydrogen in metals is needed before a critical evaluation is possible. Diffusion in certain systems such as that of hydrogen and columbium requires study over a much wider temperature range.

Initial linear rates were reported for the reaction of hydrogen with columbium.[13] Similar apparently linear reactions were noted for tantalum. However, after review of diffusion theory[14] it was concluded that such "linear" portions of the curves are fortuitous and not significant. One tantalum specimen (not activated) showed a linear reaction for 4 hours at 400 C (750 F). However, a duplicate run made with a specimen activated by heating in vacuum at 900 C (1650 F) for 1 hour gave the excellent parabolic curve seen in Figure 27. It appears that the "linear" rates are the result of interfering interface reactions.

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APPENDIX I

EFFECT OF OXYGEN AND HYDROGEN ON THE FLOW AND FRACTURE CHARACTERISTICS OF TANTALUM AND COLUMBIUM

Throughout the course of this research program, a considerable amount of data has been collected concerning the effects of temperature, and oxygen and hydrogen on the flow and fracture characteristics of tantalum and columbium. These data are included as a supplement to the discussion of Notch Sensitivity. Illustrated stress-strain curves are generally representative of two specimens tested under identical test conditions. Fracture characteristics were estimated from an examination of fracture surfaces of tested specimens at approximately 10X.

Tantalum

Effects of Oxygen

The effects of oxygen on the engineering stress-strain curves of wrought and recrystallized tantalum at room temperature are illustrated in Figure 33. Increasing the oxygen content of the wrought material increased the yield and ultimate tensile strength markedly. A yield-point discontinuity appeared when the oxygen content was raised to 758 ppm. Elongation and fracture strength did not appear to be greatly affected by oxygen content. The ductility of the recrystallized material exhibited a far greater sensitivity to oxygen content. There was a pronounced decrease in elongation and increase in fracture strength with increasing oxygen content.

The ultimate tensile strength of both the wrought and recrystallized tantalum was generally between 10,000 and 15,000 psi higher than the yield strength. This relationship was not altered by the oxygen content. However, increasing the oxygen content resulted in significant over-all strengthening and did lower the strain at maximum load.

A comparison of the room-temperature flow curves of oxygenated and pure tantalum, in the recrystallized condition, is illustrated in Figure 34. Increasing the oxygen content did not appreciably alter the work-hardening characteristics of the material.

Figure 35 illustrates the effect of temperature on the stress-strain curves of oxygenated tantalum in the recrystallized condition. The stress-strain curves of the tantalum with 489 ppm oxygen added indicate that lowering the temperature decreases the ability of the material to work harden.

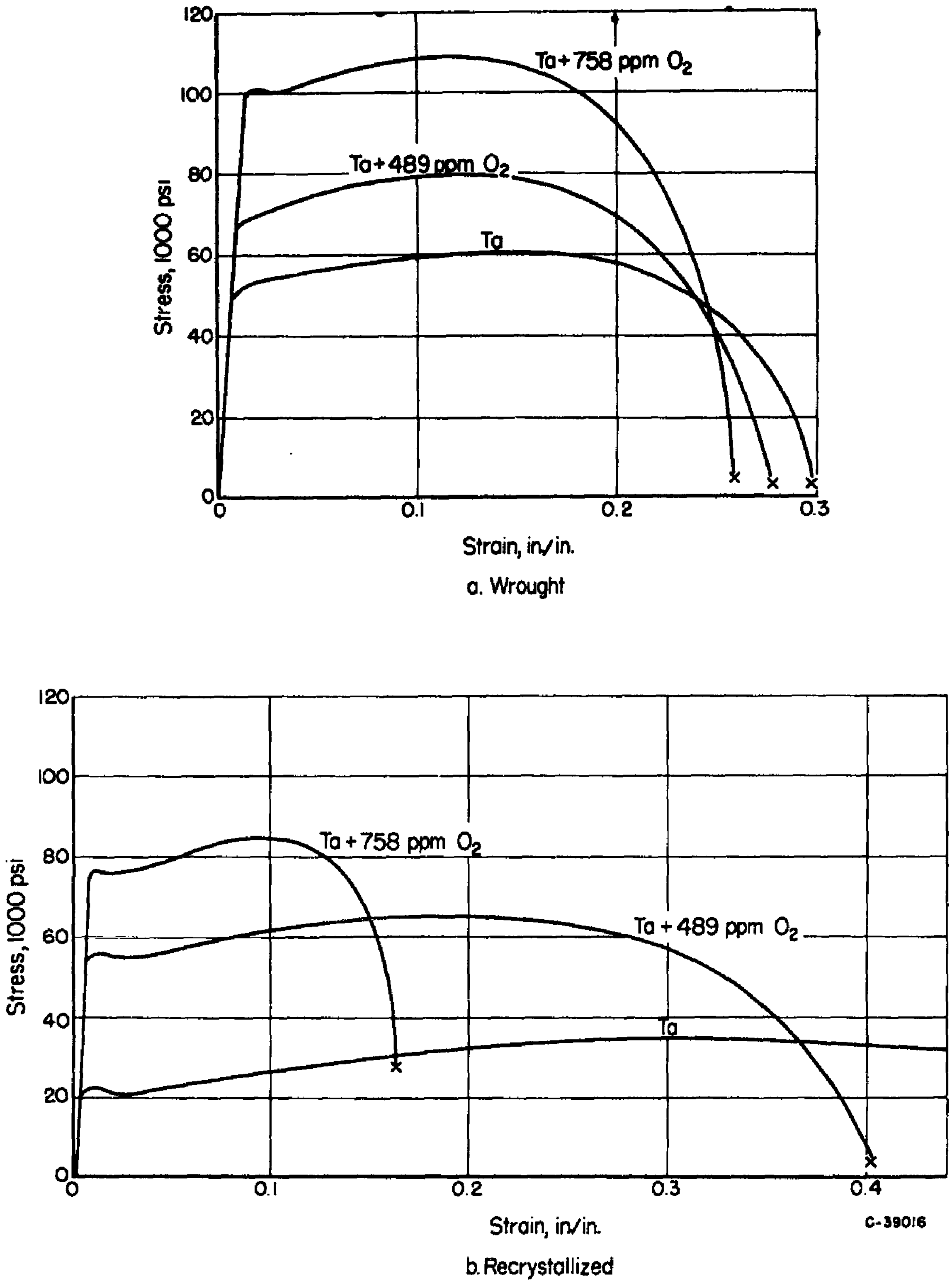

FIGURE 33. EFFECT OF OXYGEN ON THE ENGINEERING STRESS-STRAIN CURVES OF TANTALUM AT ROOM TEMPERATURE (75 F)

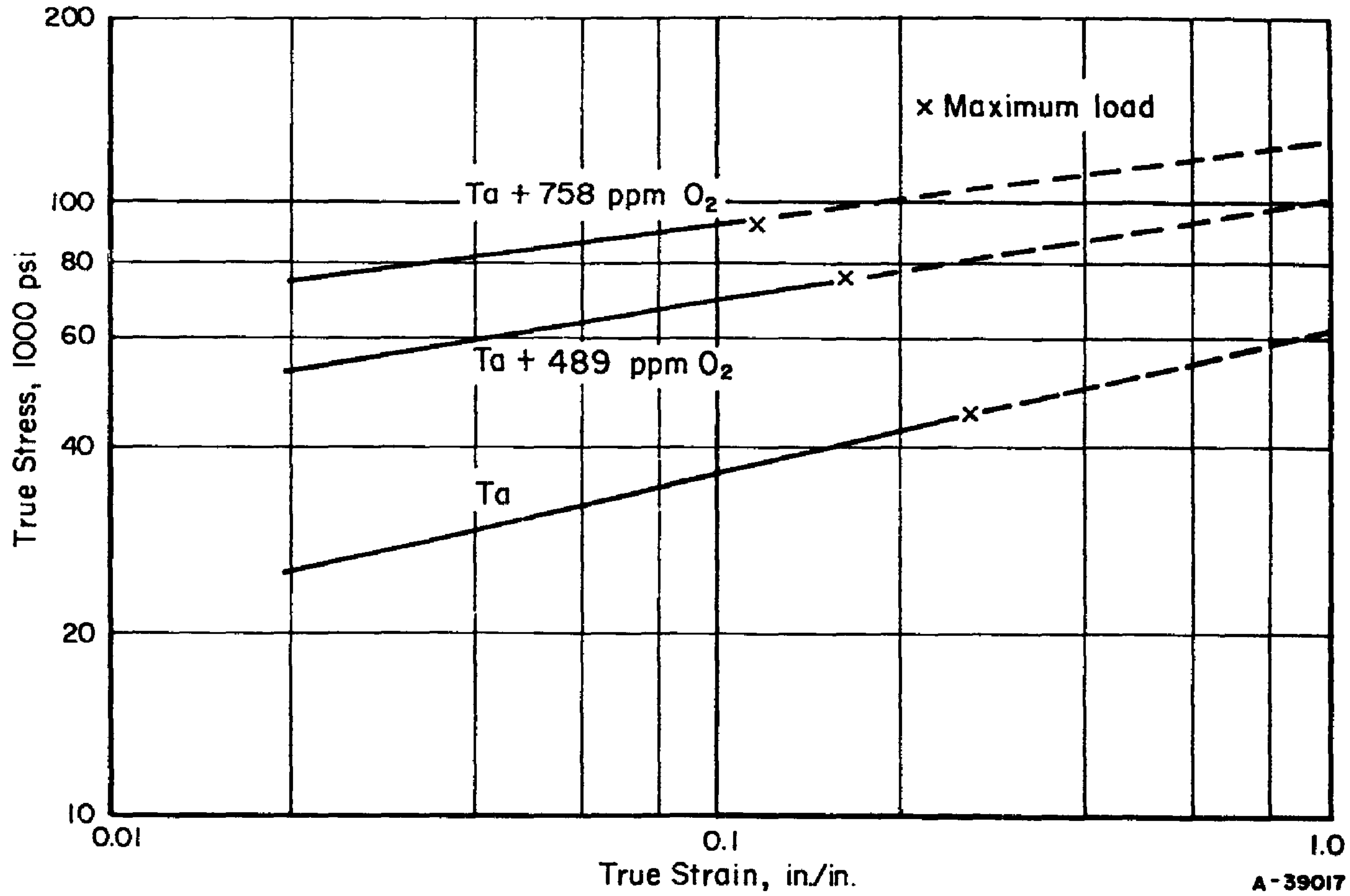

FIGURE 34. EFFECT OF OXYGEN ON THE PLASTIC-FLOW BEHAVIOR OF RECRYSTALLIZED TANTALUM AT ROOM TEMPERATURE (75 F)

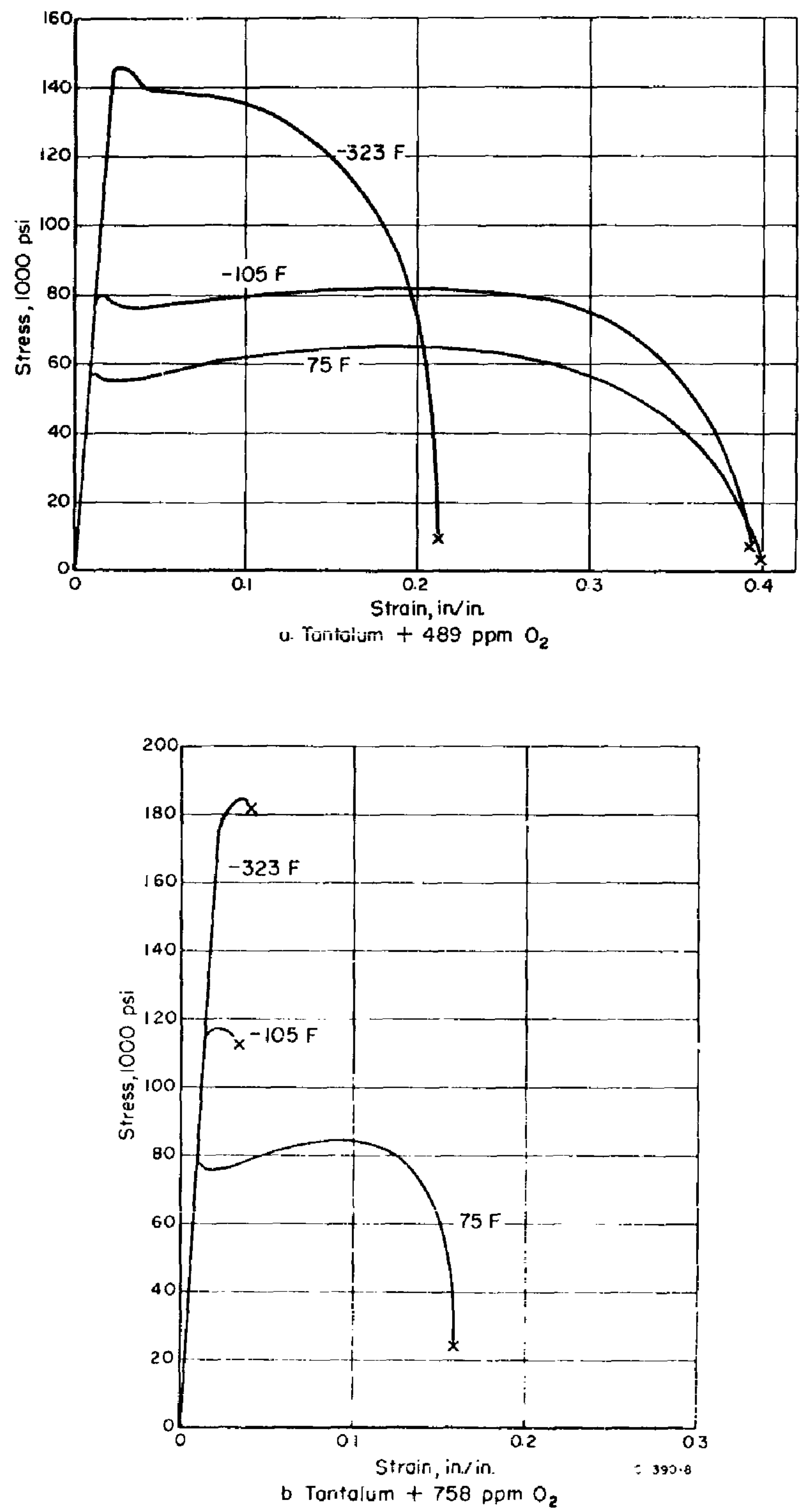

FIGURE 35. EFFECT OF TEMPERATURE ON THE ENGINEERING STRESS-STRAIN CURVE OF OXYGENATED RECRYSTALLIZED TANTALUM

At -323 F, the specimen necked immediately after yielding, and the load continuously decreased to the point of fracture. Fracture of the tantalum with 758 ppm oxygen added occurred at too low a strain to allow a similar comparison to be made with this material. However, the same effect has been observed with pure tantalum[1].

Decreasing the temperature from 75 F to -105 F increased the yield strength, but did not appreciably lower the ductility of the tantalum with 489 ppm oxygen added. However, this same temperature drop greatly lowered the ductility of the tantalum with 758 ppm oxygen added. The specimen tested at -105 F fractured shortly after yielding. Although further decreasing the temperature to -323 F increased the yield strength markedly, there was still a small amount of plastic flow before fracture. Ductility of the tantalum with 489 ppm oxygen added was appreciably lower at -323 F than at -105 F.

The effect of oxygen and temperature on the fracture characteristics of recrystallized tantalum is illustrated in Figure 36. Cleavage facets could not be detected on the fracture surface of pure tantalum at temperatures as low as -420 F. When the oxygen content was increased to approximately 489 ppm, however, indications of cleavage facets were detected at -420 F. Cleavage-type fractures predominated at -105 F and below at the highest oxygen content investigated. Barrett[20]* has observed cleavage chiefly on {110} planes in high-purity tantalum impact deformed at -323 F. Bakish[21] has reported cleavage on {110} and {100} planes in air-oxidized tantalum. A high degree of deformation is visible near the fracture surface in the illustration of the specimen with 489 ppm oxygen added and tested at 75 F. Voids are apparent and appear to be located primarily in highly deformed areas or at grain boundaries. The void density appeared to increase slightly with increasing oxygen content.

Effects of Hydrogen

Figure 37 illustrates the effect of hydrogen on the engineering stress-strain curves of wrought and recrystallized tantalum at room temperature. Increasing the hydrogen content increased the strength slightly with the most strengthening occurring in wrought material. The greatest effect of hydrogen, however, was on ductility. This is especially apparent at the "high" hydrogen level where the wrought material fractured before yielding and the recrystallized material fractured before necking at the maximum load. It appears that high hydrogen contents promote premature failure without appreciably affecting the strength.

The effect of hydrogen on the room-temperature flow properties of recrystallized tantalum is illustrated in Figure 38. It does not appear that

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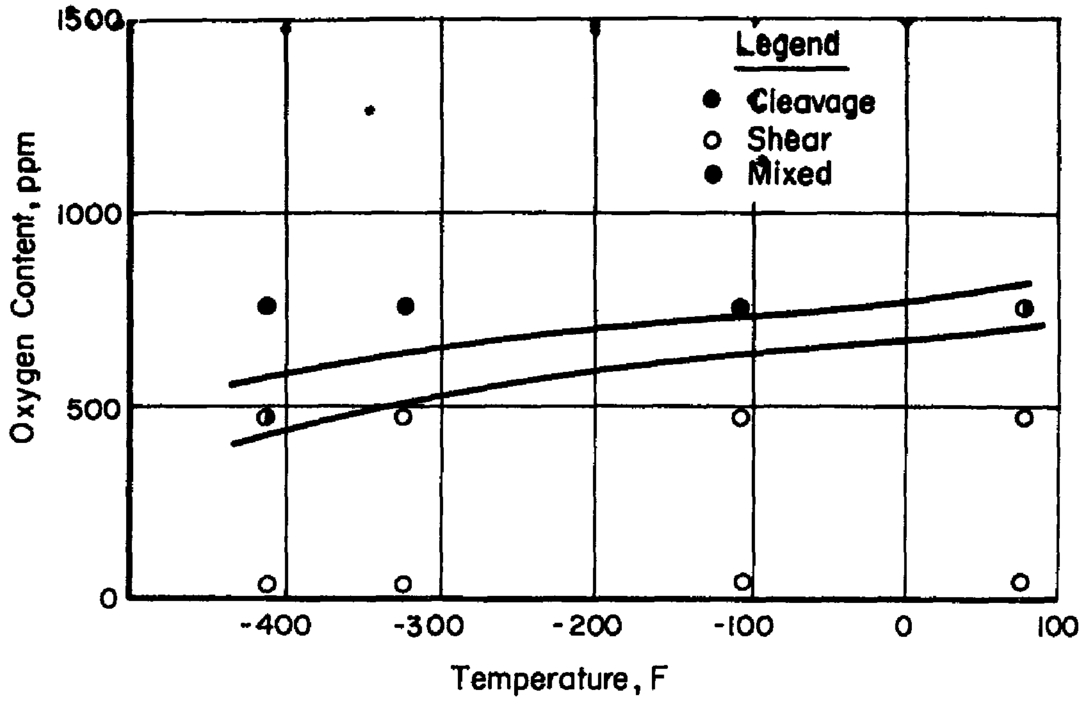
*References are given on page 75.



100X N79791

75 F

489 PPM Oxygen

FIGURE 36. EFFECT OF OXYGEN AND TEMPERATURE ON THE FRACTURE CHARACTERISTICS OF RECRYSTALLIZED TANTALUM

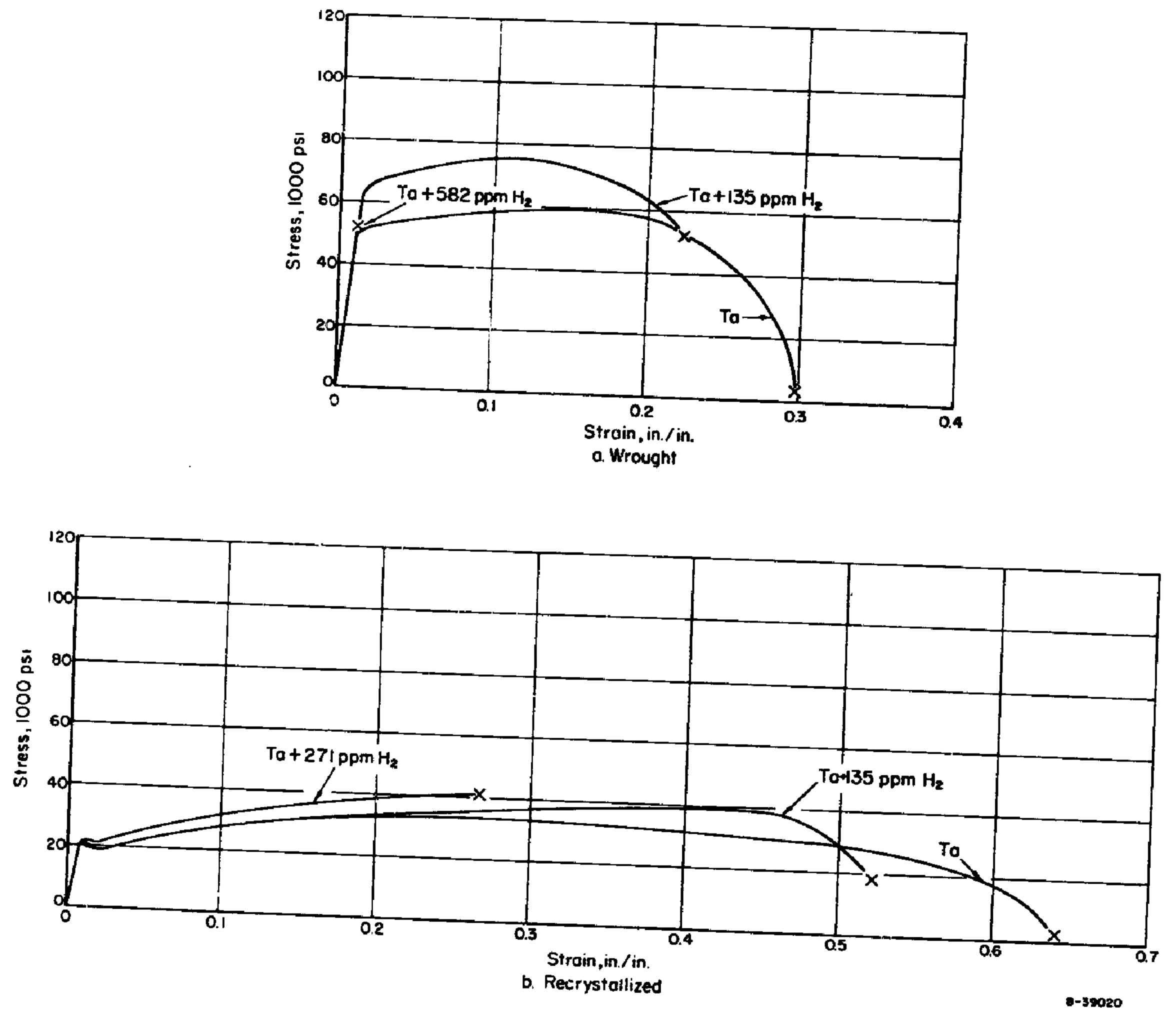


FIGURE 37. EFFECT OF HYDROGEN ON THE ENGINEERING STRESS-STRAIN CURVES OF TANTALUM AT ROOM TEMPERATURE (75 F)

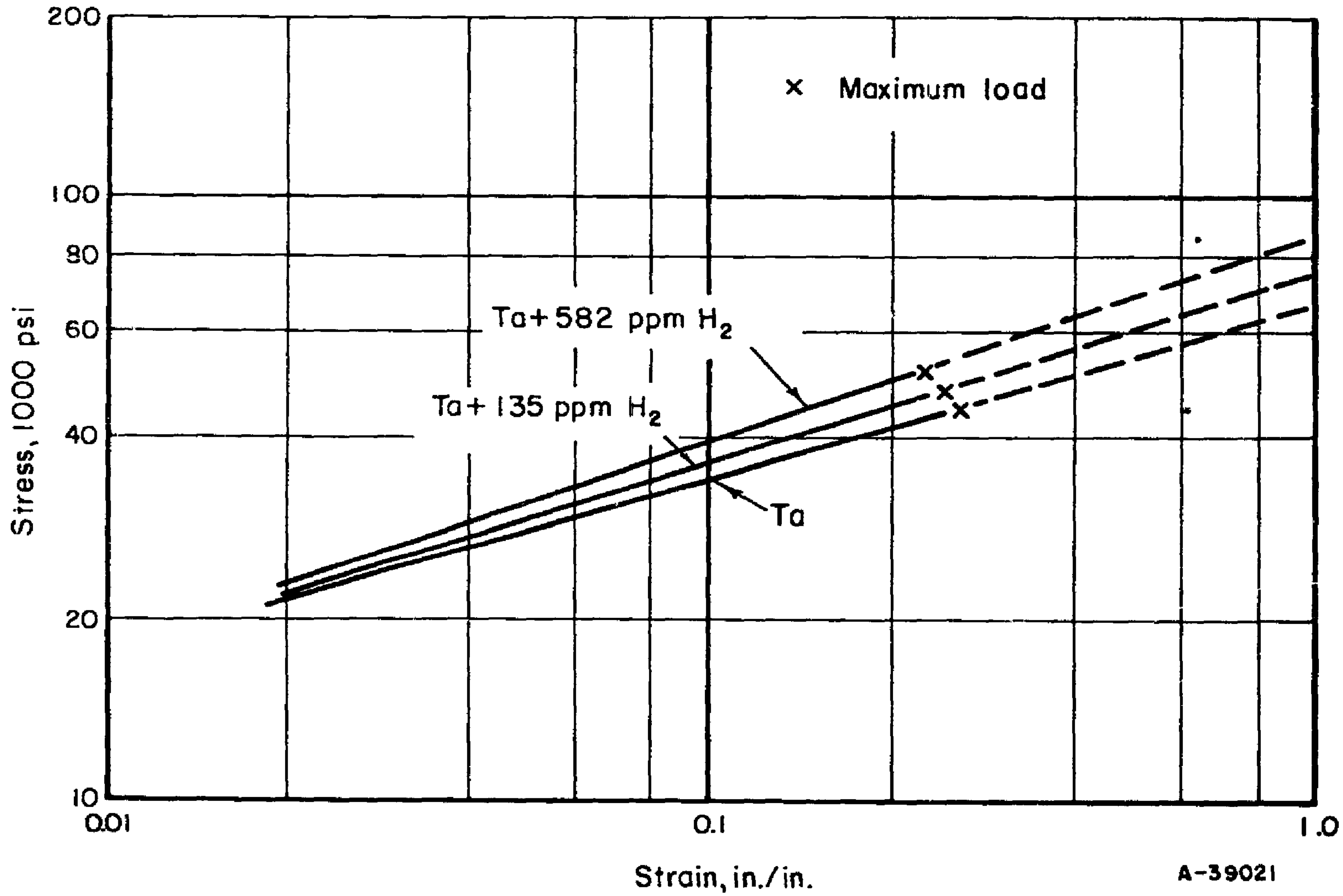


FIGURE 38. EFFECT OF HYDROGEN ON THE PLASTIC-FLOW BEHAVIOR OF RECRYSTALLIZED TANTALUM AT ROOM TEMPERATURE (75 F)

increasing the hydrogen content appreciably alters the work-hardening characteristics of tantalum.

Figure 39 illustrates the effect of temperature on hydrogenated, recrystallized tantalum. Ductilities were relatively low at -105 F, but were somewhat higher at both lower and higher temperatures. As was explained in a previous section of this report, this is thought to be an indication of a strain-aging phenomenon due to the interaction of interstitial hydrogen atoms with dislocations. Fracture occurred before other indications of strain-aging such as serrations in the stress strain curve could be detected. However, the hydrogenated material exhibited work hardening at -323 F whereas oxygenated and pure tantalum did not. This may be due to slight strain aging in hydrogenated material at -323 F. Yield points generally appeared at room temperature and below, but were not observed at elevated temperatures.

The effect of hydrogen and temperature on the fracture characteristics of recrystallized tantalum is illustrated in Figure 40. Hydrogen, in even relatively small quantities, appeared to promote brittle cleavage failure of tantalum. With approximately 300 ppm hydrogen added, failure was primarily by cleavage at room temperature. Bakish(21) has reported cleavage fracture on {100} and {110} planes in hydrogen-charged tantalum. Small transgranular cleavage cracks are visible near the fracture surface of the illustrated specimen tested at -420 F. Cleavage facets on the fracture surface are blurred due to rounding during mechanical polishing.

Columbium

Effects of Oxygen

Figure 41 illustrates the effect of oxygen on the engineering stress-strain curve of wrought and recrystallized columbium at room temperature. Increasing the oxygen content of wrought columbium to 1320 ppm oxygen increased the yield strength and ultimate tensile strength, but did not appreciably reduce ductility as measured by total elongation at fracture. However, the fracture strength did increase with increasing oxygen content. A yield point discontinuity appeared at the 1320-ppm-oxygen level.

The effects of oxygen on recrystallized columbium were not as pronounced as was observed with tantalum. At the 484-ppm level, the total elongation at fracture was considerably less than for the high-purity stock. Although increasing the oxygen content to 1320 ppm raised the fracture strength, it did not significantly lower the elongation.

The ultimate tensile strength of columbium in both structural conditions was between 10,000 and 15,000 psi higher than the yield strength.

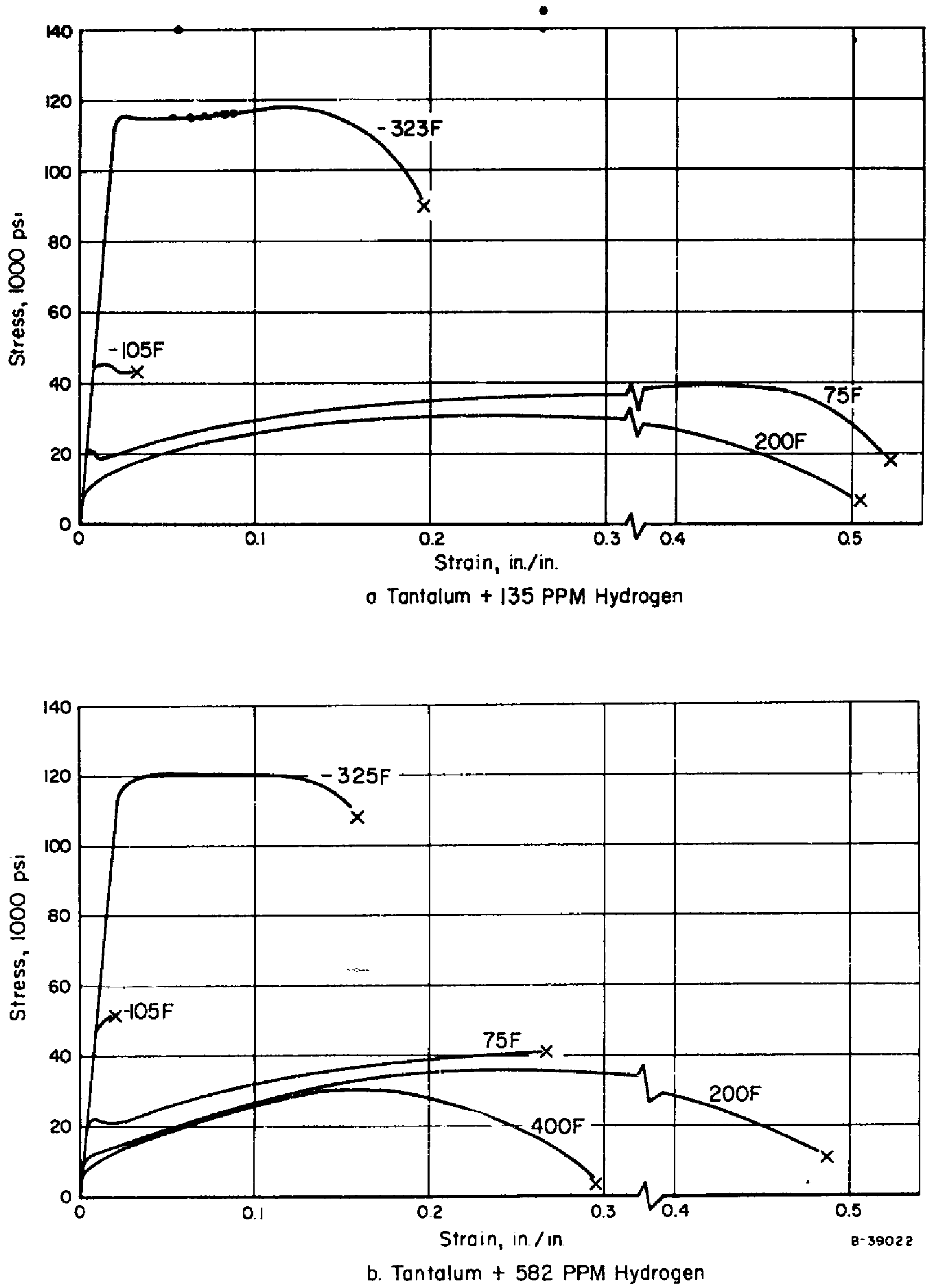
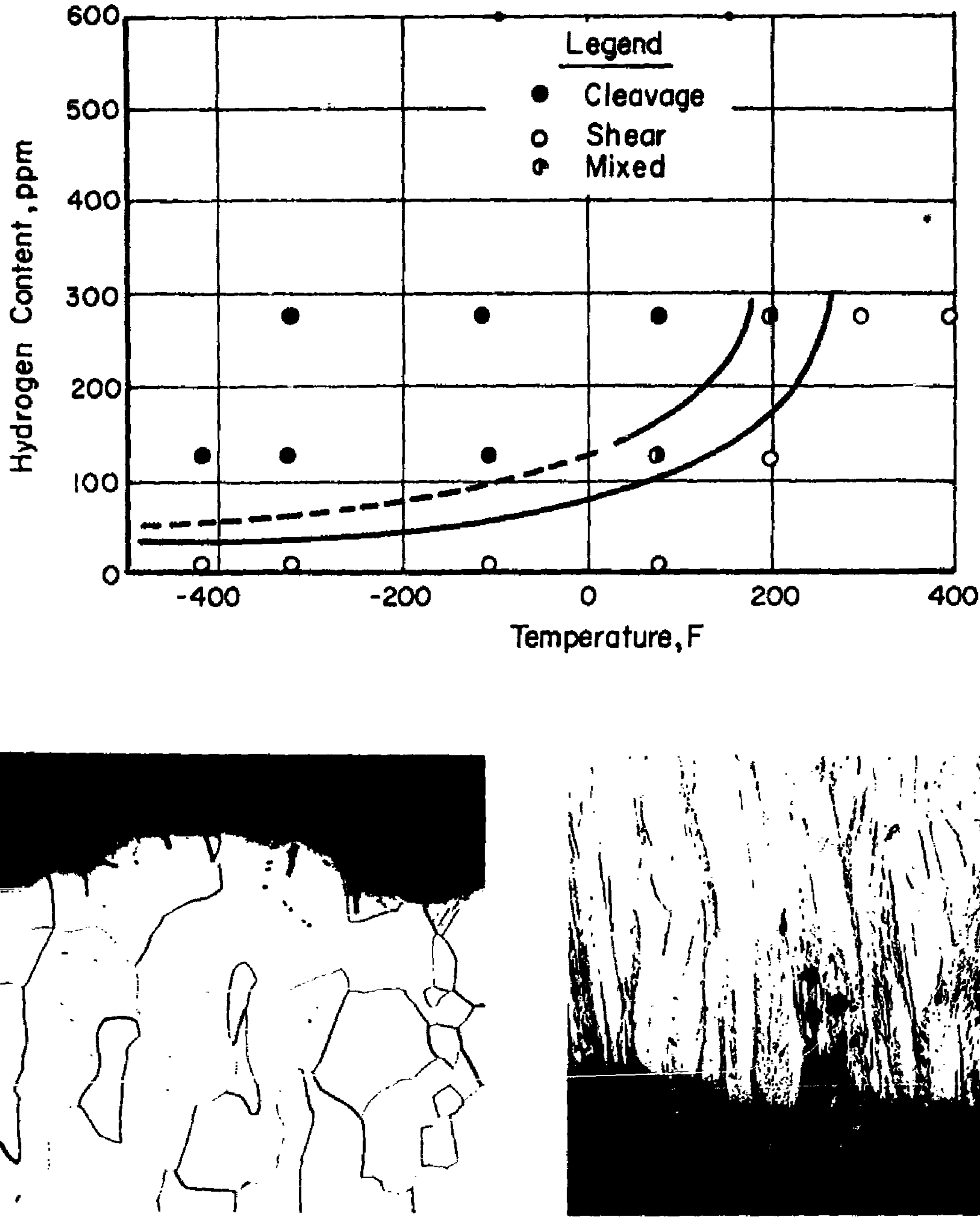


FIGURE 39. EFFECT OF TEMPERATURE ON THE ENGINEERING STRESS-STRAIN CURVES OF HYDROGENATED RECRYSTALLIZED TANTALUM



100X N81881

Shear
200 F

100X N81883

Cleavage
-420 F

135 PPM Hydrogen

FIGURE 40. EFFECT OF HYDROGEN AND TEMPERATURE ON THE FRACTURE CHARACTERISTICS OF RECRYSTALLIZED TANTALUM

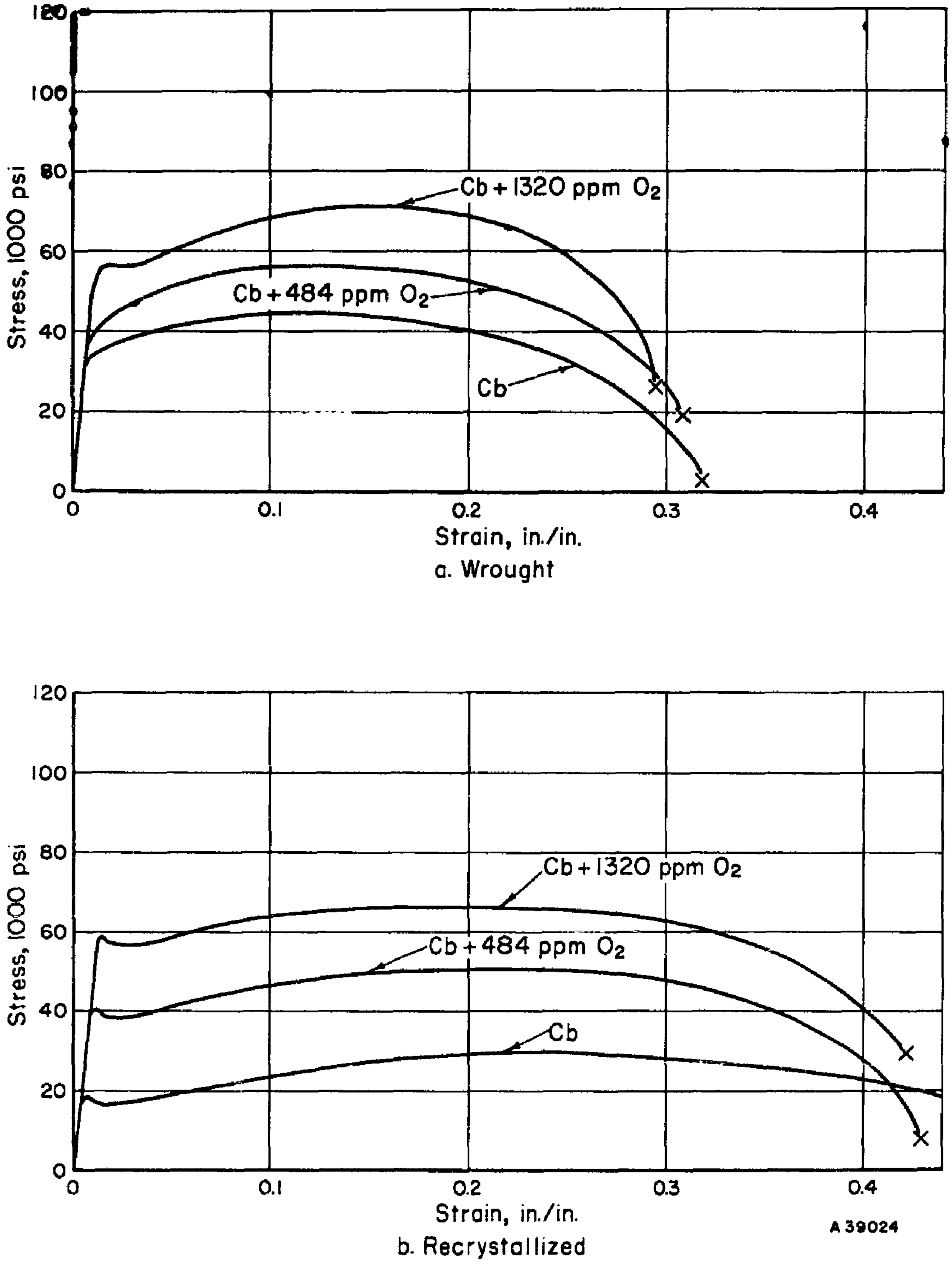


FIGURE 41. EFFECT OF OXYGEN ON THE ENGINEERING STRESS-STRAIN CURVES OF COLUMBIUM AT ROOM TEMPERATURE (75 F)

Just as with tantalum, this relationship did not appear to be altered much by increasing the oxygen content.

As shown in Figure 42, increasing the oxygen content of recrystallized columbium to 1320 ppm did not alter the work-hardening characteristics or strain at maximum load.

The effect of temperature on the engineering stress-strain curves of oxygenated columbium in the recrystallized condition is illustrated in Figure 43. Decreasing the temperature reduced the elongation at fracture and raised the fracture strength at both the 484- and 1320-ppm oxygen levels. However, these effects were more pronounced at the 1320-ppm level. Yield-point discontinuities disappeared at -323 F.

It is interesting to note that the effects of temperature on columbium with additions of approximately 500 ppm oxygen are more pronounced than those observed for tantalum at the same oxygen level. However, at the approximately 1000-ppm-oxygen level, the effect of temperature was more severe for tantalum than for columbium. Therefore, it appears that oxygen contents above approximately 500 ppm are more detrimental to the ductility and transition behavior of tantalum than of columbium.

Figure 44 illustrates the effect of oxygen and temperature on the fracture characteristics of recrystallized columbium. Increasing the oxygen content over the range studied appeared to raise the temperature below which cleavage fractures predominate. Nevertheless, failure at room temperature was still primarily by shear at the highest oxygen content studied. Similar to what was observed with oxygenated tantalum, voids were formed in highly deformed regions of ductile fractures. The void density appeared to increase somewhat with increasing oxygen content. Several transgranular cleavage cracks are visible near the fracture surface of the illustrated specimen tested at -420 F.

Effects of Hydrogen

The effect of hydrogen on the room-temperature engineering stress-strain curves of wrought and recrystallized columbium is illustrated in Figure 45. Increasing the hydrogen content of columbium increased the strength of wrought and recrystallized structures to a somewhat greater extent than was observed with tantalum. The primary effect of hydrogen is that it apparently promotes premature failure, severely reducing the ductility. This effect is more pronounced than was observed with tantalum.

The effect of hydrogen on the room-temperature flow properties of recrystallized columbium is illustrated in Figure 46. Increasing the hydrogen content does not appreciably alter the work-hardening characteristics of

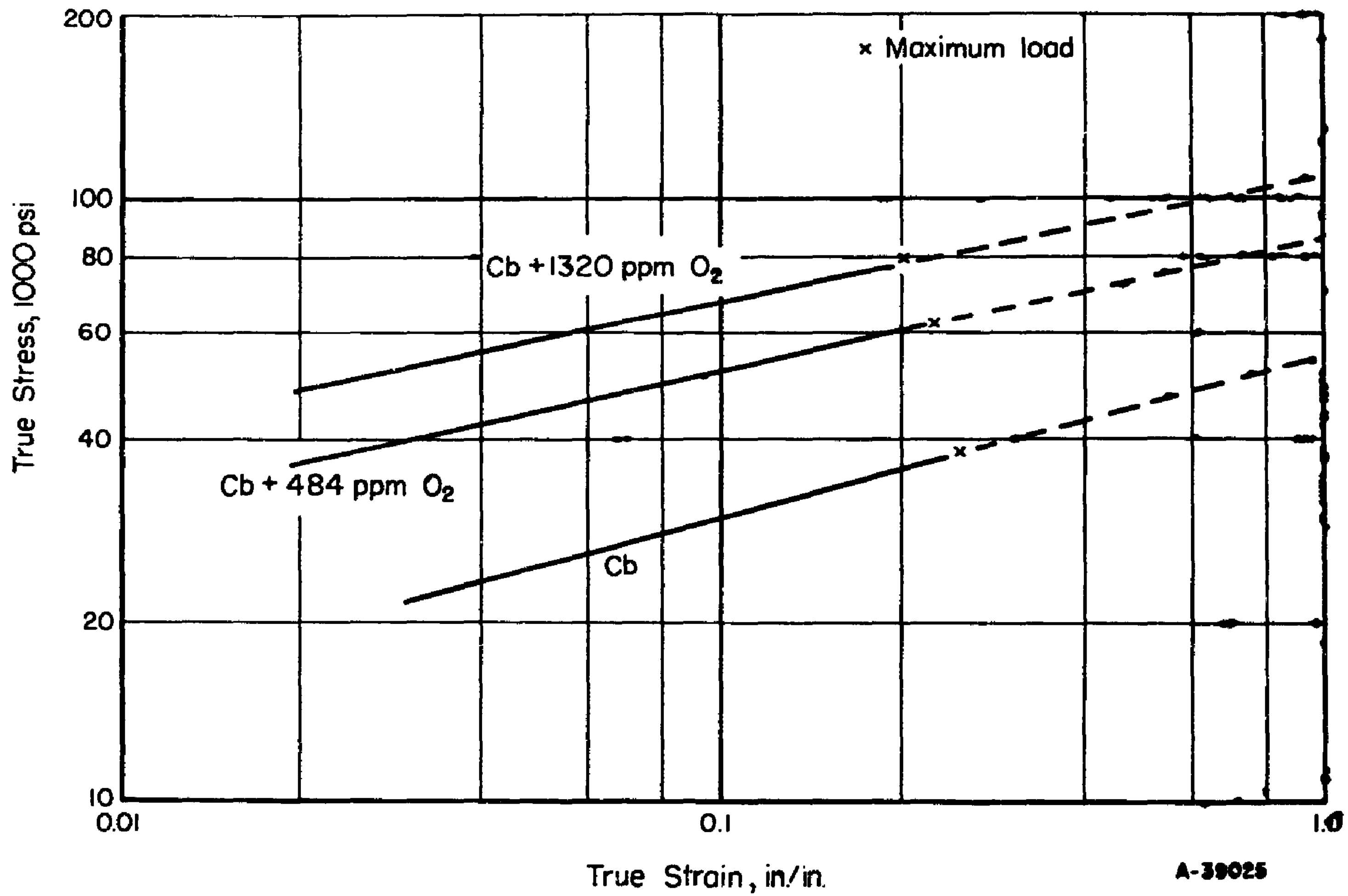
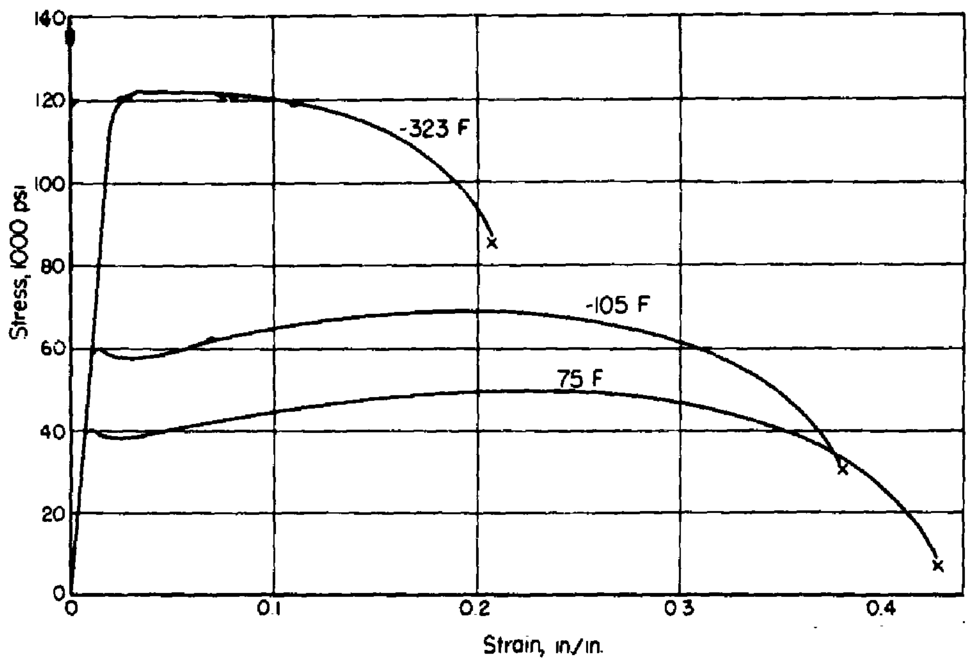
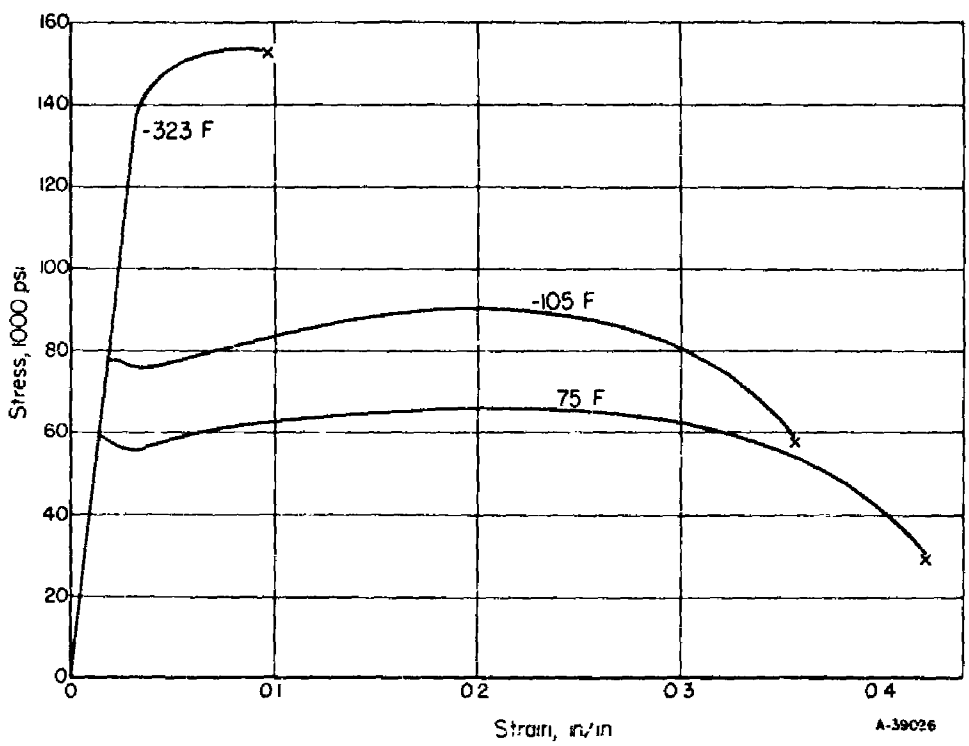


FIGURE 42. EFFECT OF OXYGEN ON THE PLASTIC-FLOW BEHAVIOR OF RECRYSTALLIZED COLUMBIUM AT ROOM TEMPERATURE (75 F)

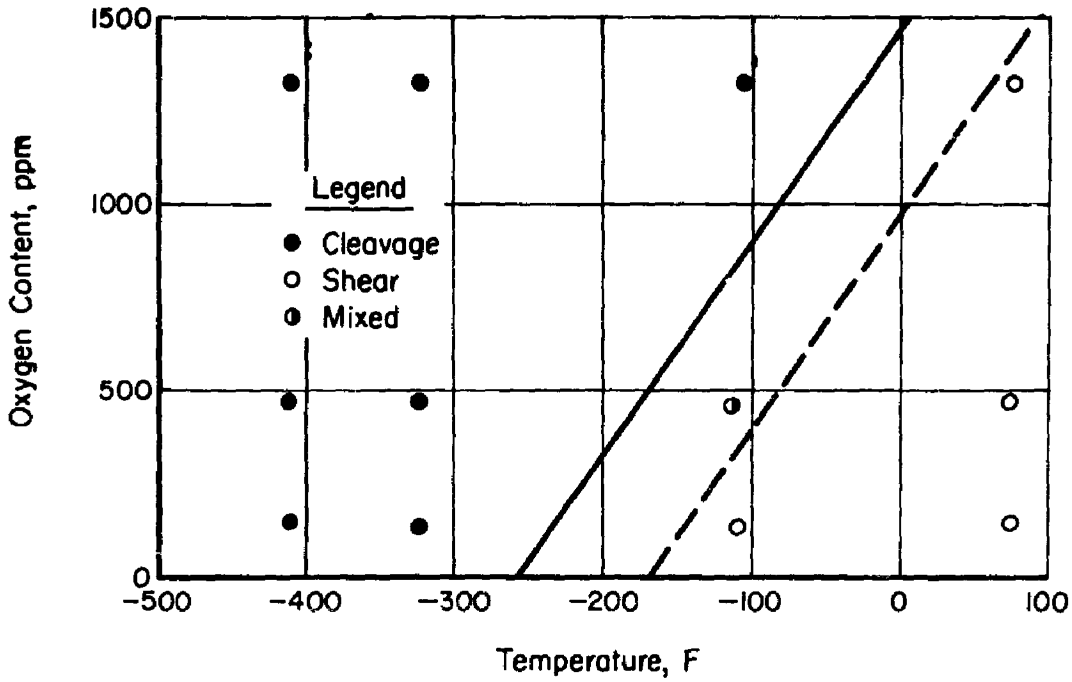


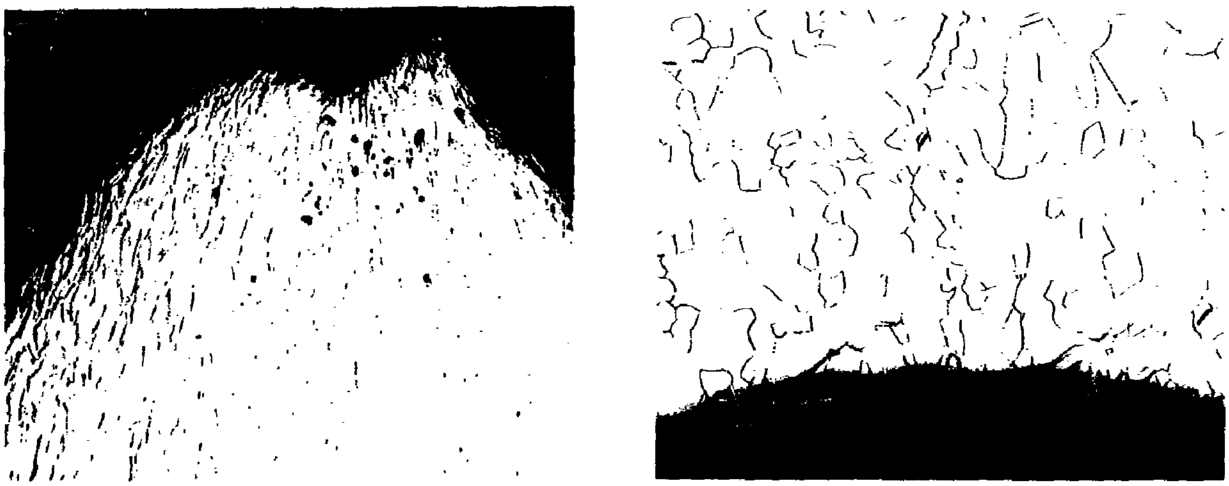
a Columbium + 484 PPM O_2



b Columbium + 1320 PPM O_2

FIGURE 43. EFFECT OF TEMPERATURE ON THE ENGINEERING STRESS-STRAIN CURVES OF OXYGENATED RECRYSTALLIZED COLUMBIUM





100X N79780

Shear
75 F

100X N81876

Cleavage
-420 F

484 PPM Oxygen

FIGURE 44. EFFECT OF OXYGEN AND TEMPERATURE ON THE FRACTURE CHARACTERISTICS OF RECRYSTALLIZED COLUMBIUM

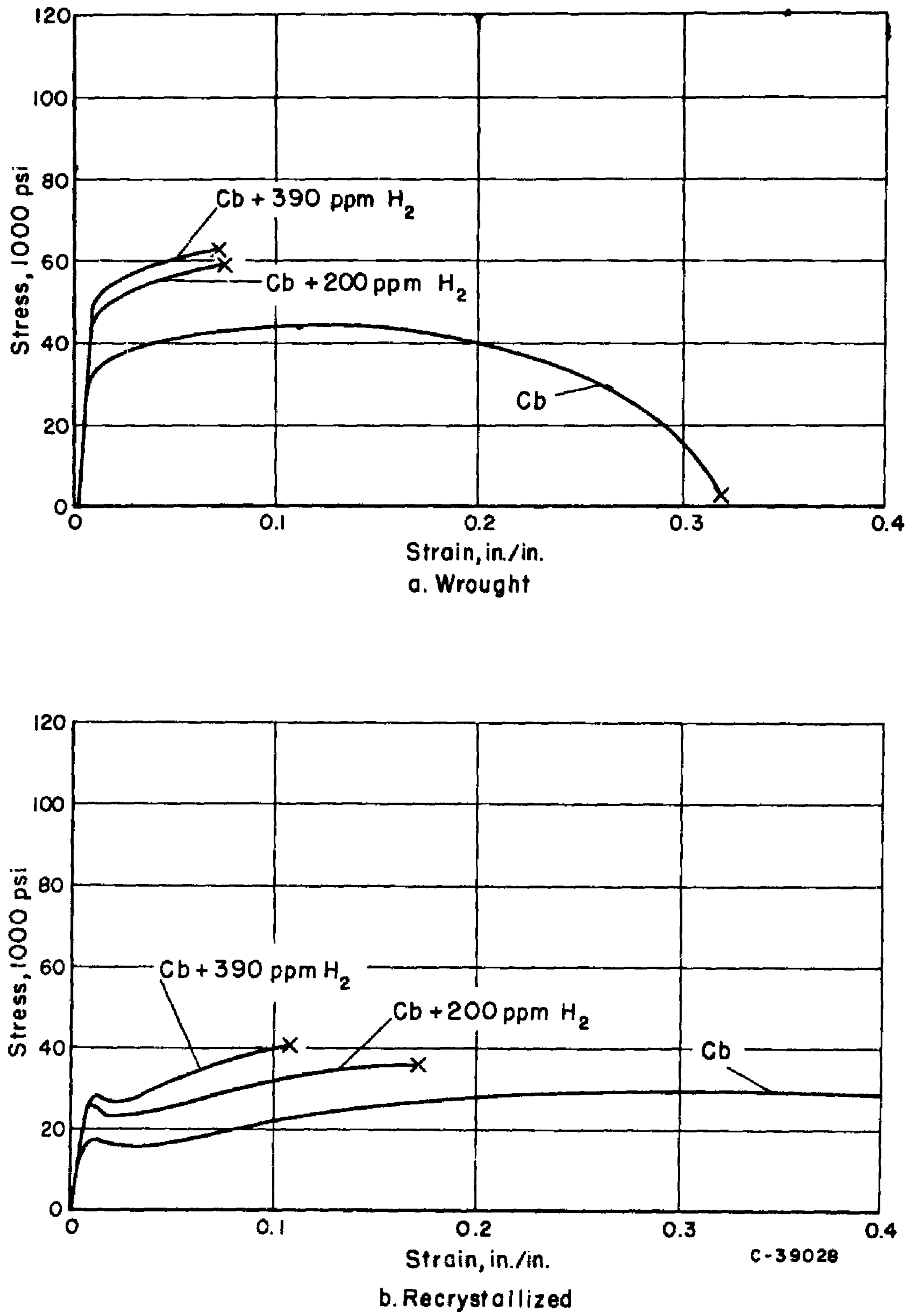


FIGURE 45. EFFECT OF HYDROGEN ON THE ENGINEERING STRESS-STRAIN CURVES OF COLUMBIUM AT ROOM TEMPERATURE

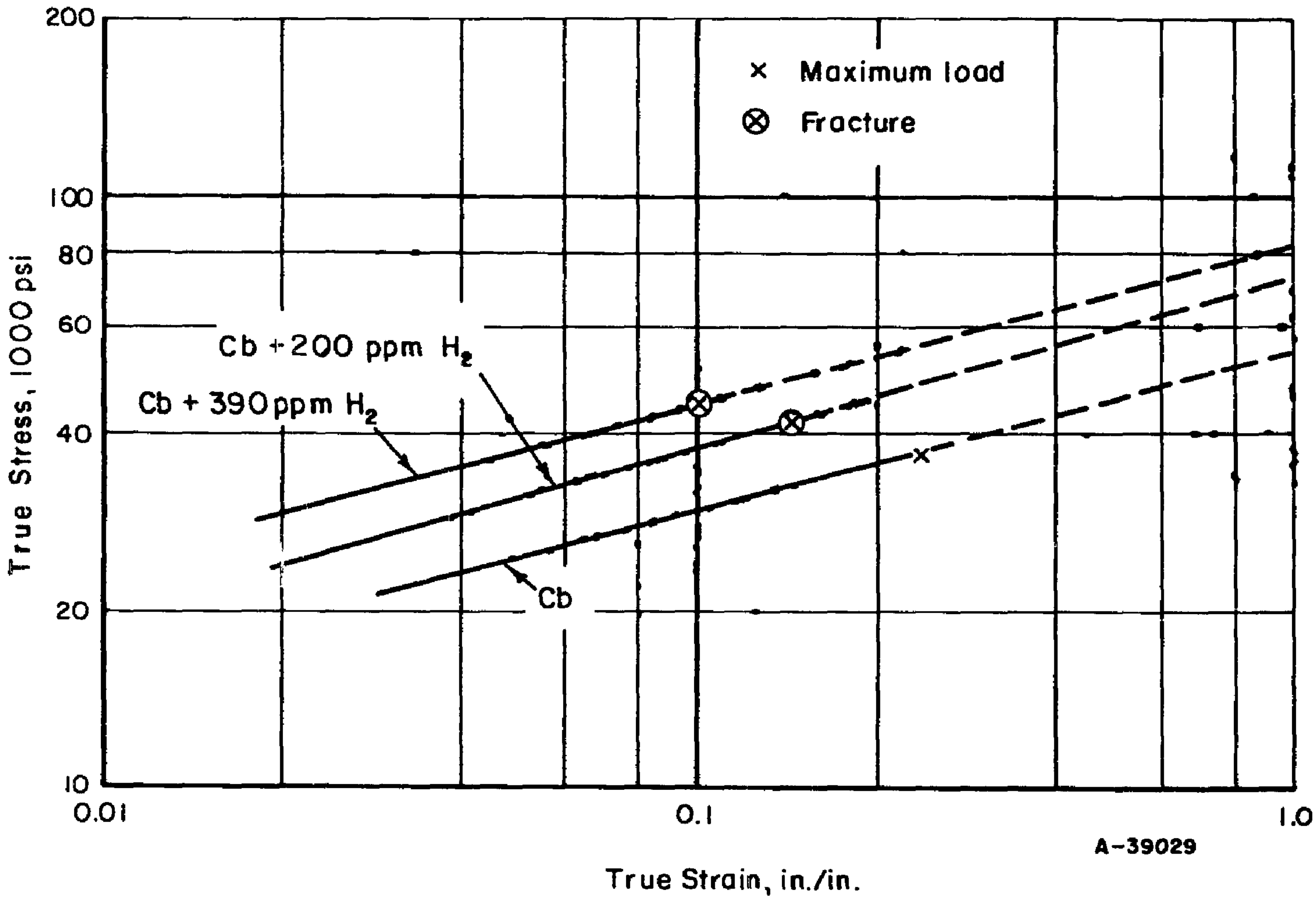
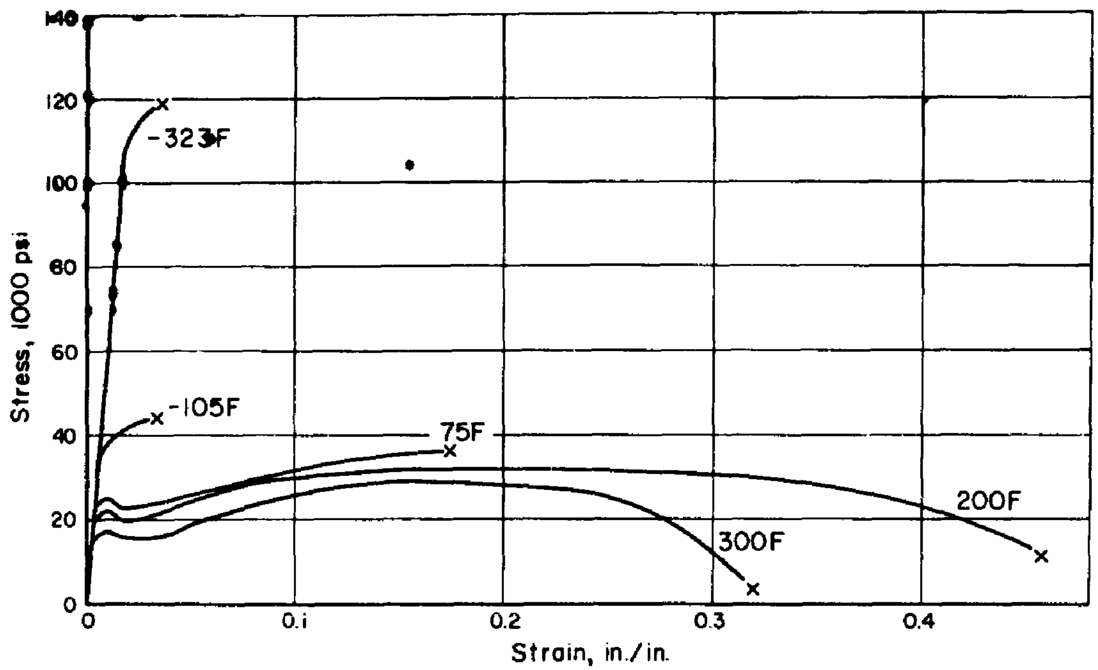


FIGURE 46. EFFECT OF HYDROGEN ON THE PLASTIC-FLOW BEHAVIOR OF RECRYSTALLIZED COLUMBIUM AT ROOM TEMPERATURE (75 F)

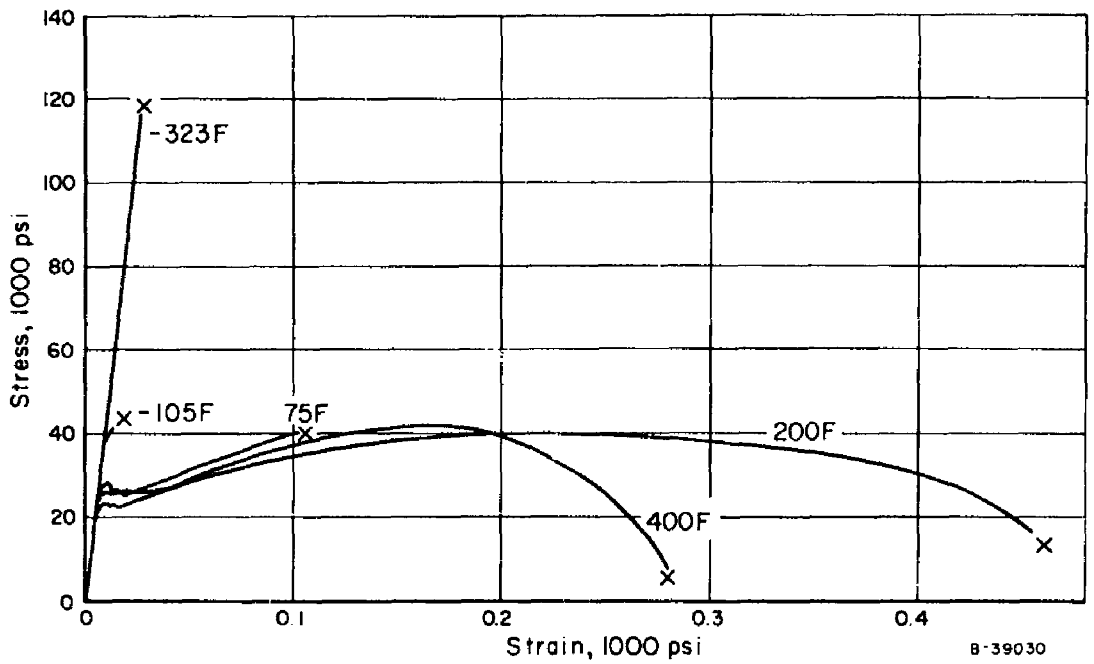
columbium. However, the strain at maximum load does appear to decrease significantly with increasing hydrogen. This is probably due largely to premature failure of hydrogenated specimens.

Figure 47 illustrates the effect of temperature on the engineering stress-strain curve of hydrogenated, recrystallized columbium. Behavior at both hydrogen levels was similar. At -323 and -105 F fracture occurred at or just after yielding. Specimens tested at 75 F appeared to fail prematurely before necking. Increasing the test temperature above 200 F decreased the total elongation at fracture.

Figure 48 illustrates the effect of hydrogen and temperature on the fracture characteristics of recrystallized columbium. Pure columbium failed predominately by cleavage at -323 F and below, whereas cleavage fractures predominated for hydrogenated columbium tested at room temperature and below. Numerous transgranular cleavage cracks can be seen near the fracture surface of the illustrated specimen tested at -420 F.

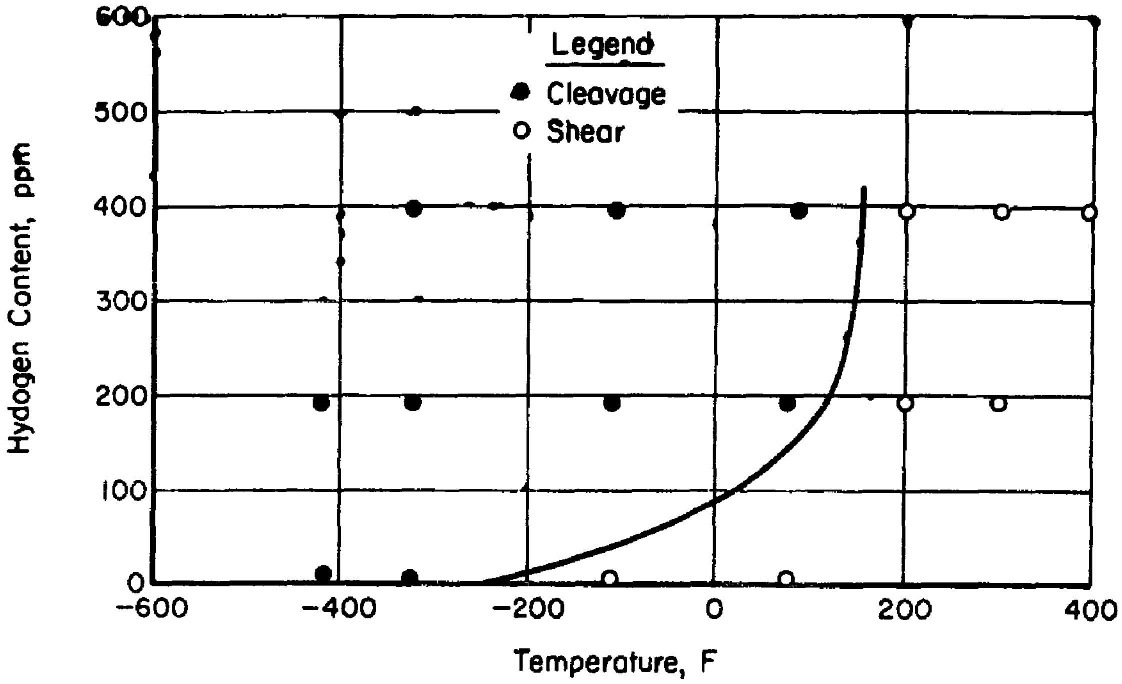


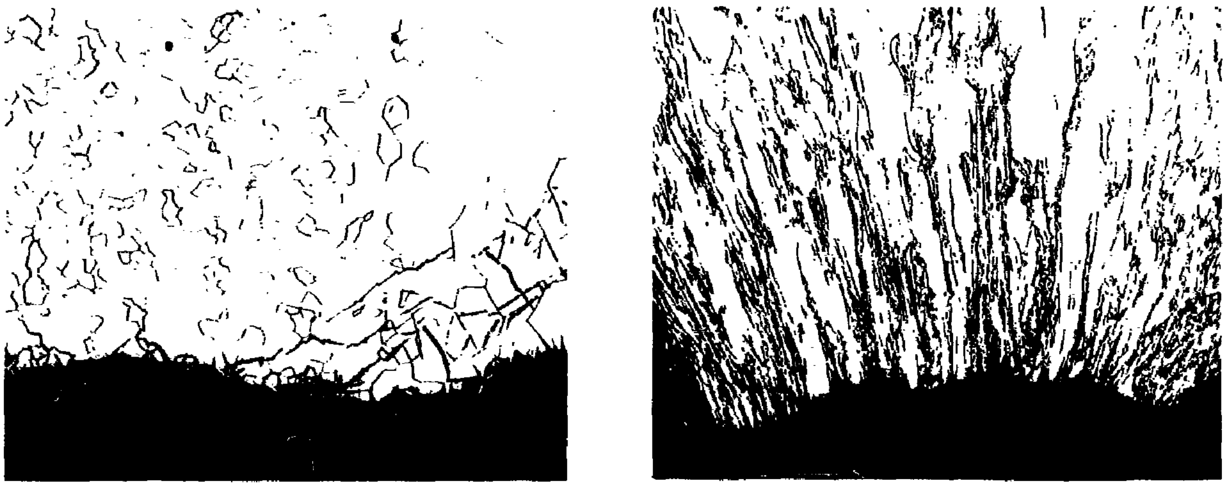
a. Columbium + 200 PPM Hydrogen



b. Columbium + 390 PPM Hydrogen

FIGURE 47. EFFECT OF TEMPERATURE ON THE ENGINEERING STRESS-STRAIN CURVES OF HYDROGENATED RECRYSTALLIZED COLUMBIUM





100X N81878 100X N81879

200 F -420 F

200 PPM Hydrogen

FIGURE 48. EFFECT OF HYDROGEN AND TEMPERATURE ON THE FRACTURE CHARACTERISTICS OF RECRYSTALLIZED COLUMBIUM

APPENDIX II

TABULATED TENSILE DATA

TABLE 16. TENSILE AND NOTCH TENSILE PROPERTIES OF WROUGHT, STRESS-RELIEVED TANTALUM WITH 500 PPM OXYGEN ADDED

Test Temperature, F	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent	Fracture at Maximum Load	Fracture Mechanism
		Unnotched Specimens				
75	71,400	82,900	28	90.6	No	Shear
75	71,600	80,000	28	91.0	No	"
-323	164,600	165,100	16	89.7	No	"
-323	161,400	165,700	20	90.4	No	Shear and cleavage
-105	89,000	92,300	28	98.4	No	Shear
-105	92,100	94,700	30	98.5	No	"
-423	191,000	192,000	16	57	No	Shear and cleavage
		Notched Specimens				
75		129,100		92.5	No	Shear
75		127,000		92.5	No	"
75		137,200		87.2	No	"
-323		247,100		40.8	No	Shear and cleavage
-323		249,300		39.9	No	Ditto
-323		250,000		75.0	No	"
-105		164,600		96.3	No	Shear
-105		157,600		97.4	No	"
-105		162,000		97.1	No	"
-423		304,000		34.7	No	Shear and cleavage

TABLE 17. TENSILE AND NOTCH TENSILE PROPERTIES OF RECRYSTALLIZED TANTALUM WITH 500 PPM OXYGEN ADDED

Test Temperature, F	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent	Fracture at Maximum Load	Fracture Mechanism
			Unnotched Specimens			
75	57,000	64,000	42	90.3	No	Shear
75	55,100	62,100	40	94.8	No	"
-323	147,000	148,800	18	82.9	No	Shear and cleavage
-323	145,700	148,300	24	71.9	No	Shear
-105	75,300	76,300	40	97.9	No	"
-105	80,900	83,200	46	97.7	No	Shear and cleavage
-423	164,000	168,000	16	58	No	Ditto
			Notched Specimens			
75		94,400		92.9	No	Shear
75		95,800		93.9	No	"
75		96,800		93.4	No	"
-323		217,700		66.3	No	"
-323		226,100		45.5	No	Shear and cleavage
-323		216,800		62.0	No	Ditto
-105		131,500		95.9	No	Shear
-105		125,300		96.1	No	Shear and cleavage
-105		130,900		97.7	No	Shear
-423		298,000		22.6	No	Shear and cleavage

TABLE 18. TENSILE AND NOTCH TENSILE PROPERTIES OF WROUGHT STRESS-RELIEVED TANTALUM WITH 1000 PPM OXYGEN ADDED

Test Temperature, F	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent	Fracture at Maximum Load	Fracture Mechanism
			Unnotched Specimens			
75	101,100	109,000	28	72.4	No	Shear
-323	192,000	192,800	12	50.0	No	Shear and cleavage
-105	111,300	113,200	32	91.0	No	Ditto
-423	215,000	221,000	6	16	No	Cleavage
			Notched Specimens			
75		181,400		92.1	No	Shear
-323		287,800		Nil	Yes	Shear and cleavage
-105		197,300		33.2	No	Ditto
-105		185,500		88.0	No	Shear
-423		261,000		0	Yes	Cleavage

TABLE 19. TENSILE AND NOTCH TENSILE PROPERTIES OF RECRYSTALLIZED TANTALUM WITH 1000 PPM OXYGEN ADDED

Test Temperature, F	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent	Fracture at Maximum Load	Fracture Mechanism
			Unnotched Specimens			
75	77,600	84,400	16	31.0	No	Shear and cleavage
-323	183,100	188,900	4	3.5	No	Cleavage
-105	116,100	116,200	2[a]	3.2	No	"
-423	211,000	218,000	4	0	Yes	"
			Notched Specimens			
75		136,400		44.6	No	Shear and cleavage
75		138,100		43.9	No	Ditto
-323		273,200		7.9	Yes	Cleavage
-323		267,700		3.2	Yes	"
-105		179,100		7.8	No	"
-105		189,600		17.4	No	"
-423		304,000		9.2	Yes	"

(a) Fracture outside 1/2-inch gage mark.

TABLE 20. TENSILE AND NOTCH TENSILE PROPERTIES OF WROUGHT, STRESS-RELIEVED TANTALUM WITH 150 PPM HYDROGEN ADDED

Test Temperature, F	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent	Fracture at Maximum Load	Fracture Mechanism
			Unnotched Specimens			
75	69,800	79,800	24	47.6	No	Shear and cleavage
75	66,200	75,900	28	59.5	No	Ditto
-323	164,800	168,700	16	30.9	No	"
-323	165,200	171,100	12	34.9	No	"
-105	87,300	87,300	2[a]	2.9	Yes	"
200	60,100	65,900	26	87.7	No	Shear and cleavage
200	63,200	68,900	24	83.8	No	Ditto
-105	73,200	80,100	2[a]	3.2	Yes	Clevage
-423	210,000	211,000	12	29.4	Yes	"
				9.4		
			Notched Specimens			
75		109,700		29.4	No	Shear and cleavage
75		107,900		29.4	No	Ditto
75		105,500		22.8	No	"
-323		250,000		11.0	No	Cleavage
-323		256,600		19.9	No	"
-323		255,900		24.0	No	"
-105		55,900		3.2	Yes	"
-105		73,800		0	Yes	"
-105		47,300		3.2	Yes	"
200		98,500		84.6	No	Shear
200		101,200		81.6	No	"
200		98,300		83.1	No	"
-423		315,000		18.5	No	Cleavage

(a) Fracture on 1/2-inch gage mark.

TABLE 21. TENSILE AND NOTCH TENSILE PROPERTIES OF RECRYSTALLIZED TANTALUM WITH 150 PPM HYDROGEN ADDED

Test Temperature, F	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent	Fracture at Maximum Load	Fracture Mechanism
			Unnotched Specimens			
75	21,000	38,200	52	53.6	No	Shear and cleavage
75	20,000	39,200	54	50.2	No	Ditto
-323	114,700	116,900	24	51.9	No	Cleavage
-323	121,700	128,000	4(a)	55.8	No	Shear and cleavage
-105	46,700	46,900	2(b)	3.8	No	Cleavage
-105	46,500	46,800	2(b)	1.9	No	"
200	12,300	32,100	52	87.6	No	Shear
200	12,100	32,300	62	87.4	No	"
-423	(c)	132,000	3	1	Yes	Cleavage
			Notched Specimens			
75		54,900		22.6	No	Cleavage
75		56,300		31.0	No	"
75		56,800		28.3	No	"
-323		201,300		25.3	No	Shear and cleavage
-323		192,300		15.3	No	Ditto
-323		207,800		21.1	No	"
-105		42,400		7.9	Yes	Cleavage
-105		58,800		0	Yes	"
-105		59,000		1.5	Yes	
200		49,800		81.6	No	"
200		50,400		81.3	No	Shear and cleavage
200		50,700		75.8	No	Ditto
-423		220,000		0	Yes	Cleavage

(a) Fracture outside 1/2-inch gage mark.

(b) Fracture on 1/2-inch gage mark.

(c) Fracture before 0.2 per cent yield.

TABLE 22. TENSILE AND NOTCH TENSILE PROPERTIES OF WROUGHT, STRESS-RELIEVED TANTALUM WITH 500 PPM HYDROGEN ADDED

Test Temperature, F	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent	Failure at Maximum Load	Fracture Mechanism
			Unnotched Specimens			
75	(a)	50,000	0(b)	0	Yes	Cleavage
-323	(a)	186,500	0(b)	0	Yes	"
-323	(a)	194,800	0(b)	0	Yes	"
-105	(a)	30,000	0(b)	0	Yes	"
-105	(a)	56,900	0(b)	0	Yes	"
300	48,500	57,900	30	77.8	No	Shear and cleavage
200	51,700	62,700	32	61.9	No	Ditto
400	48,900	56,300	26	80.8	No	"
						"
			Notched Specimens			"
75		21,700		0	Yes	Cleavage
75		12,200		0	Yes	"
-323		130,100		0	Yes	"
-105		29,200		0	Yes	"
300		84,400		47.0	No	Shear and cleavage
300		90,800		50.4	No	Ditto
200		80,500		18.1	No	Cleavage
400		70,900		72.9	No	Shear

(a) Fracture before 0.2 per cent yield.
(b) Fracture on 1/2-inch gage mark.

TABLE 23. TENSILE AND NOTCH TENSILE PROPERTIES OF RECRYSTALLIZED TANTALUM WITH 500 PPM HYDROGEN ADDED

Test Temperature, F	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent	Failure at Maximum Load	Fracture Mechanism
			Unnotched Specimens			
75	22,300	42,600	26	26.9	No	Cleavage
-323	119,000	122,200	16	25.9	No	"
-105	51,100	52,100	0	0.6	Yes	"
300	12,600	31,900	50[a]	84.6	No	Shear
200	12,100	36,000	50	82.2	No	Shear and cleavage
400	10,300	31,100	30	92.0	No	Shear
			Notched Specimens			
75		44,900		4.8	No	Cleavage
75		39,400		9.4	No	"
-323		115,600		0	Yes	"
-323		80,400		0	Yes	"
-105		44,700		0	Yes	"
-105		10,100		0	Yes	"
-105		44,200		0	Yes	"
300		49,700		73.6	No	Shear
300		49,200		67.7	No	Shear and cleavage
200		54,800		24.0	No	Cleavage
400		46,900		93.0	No	Shear

(a) Fracture on 1/2-inch gage mark.

TABLE 24. TENSILE AND NOTCH TENSILE PROPERTIES OF WROUGHT, STRESS-RELIEVED COLUMBIUM WITH 500 PPM OXYGEN ADDED

Test Temperature, F	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent	Fracture at Maximum Load	Fracture Mechanism
			Unnotched Specimens			
75	45,200	59,700	34	83.9	No	Shear
75	45,800	60,600	32	85.5	No	Shear and cleavage
-323	128,500	134,000	14	60.0	No	Cleavage
-323	120,100	122,800	14	70.8	No	"
-105	59,100	74,500	30	71.8	No	Shear and cleavage
-105	67,100	83,500	28	66.1	No	Shear
-423	196,000	204,000	4	7	--	Cleavage
			Notched Specimens			
75		107,400		80.4	No	
75		106,700		80.1	No	Shear and cleavage
75		95,300		86.5	No	Ditto
-323		208,300		7.1	Yes	Cleavage
-323		204,000		22.4	No	"
-323		201,600		26.5	No	"
-105		134,700		74.2	No	"
-105		116,300		84.8	No	"
-105		125,300		81.6	No	"
-423		209,000		0	Yes	"

TABLE 25. TENSILE AND NOTCH TENSILE PROPERTIES OF RECRYSTALLIZED COLUMBIUM WITH 500 PPM OXYGEN ADDED

Test Temperature, F	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent	Fracture at Maximum Load	Fracture Mechanism
			Unnotched Specimens			
75	36,400	49,200	46	88.9	No	Shear
75	42,600	50,600	44	91.0	No	"
-323	121,400	125,700	22	58.9	No	Cleavage
-323	131,500	139,300	22	42.7	No	"
-105	68,500	73,800	36	67.7	No	Shear
-105	55,500	64,200	42	75.0	No	Shear and cleavage
-423	168,000	179,000	1	1	Yes	Cleavage
			Notched Specimens			
75		85,800		87.2	No	Shear and cleavage
75		86,800		91.3	No	Shear
75		90,100		87.8	No	"
-323		182,800		0	Yes	Cleavage
-323		182,800		0	Yes	"
-323		216,800		4.7	Yes	"
-105		101,100		70.4	No	Shear
-105		113,100		65.5	No	Shear and cleavage
-105		114,500		62.6	No	Ditto
-423		169,000		0	Yes	Cleavage
-423		201,000		3.1	Yes	"

TABLE 26. TENSILE AND NOTCH TENSILE PROPERTIES OF WROUGHT STRESS-RELIEVED COLUMBIUM WITH 1000 PPM OXYGEN ADDED

Test Temperature, F	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent	Fracture at Maximum Load	Fracture Mechanism
			Unnotched Specimens			
75	61,200	75,300	34	84.8	No	Shear and cleavage
75	65,700	78,500	36	82.9	No	Ditto
-323	151,000	157,600	20	57.3	No	Cleavage
-323	155,000	159,900	22	58.4	No	"
-105	77,800	86,900	38	79.0	No	Shear and cleavage
-105	84,700	98,400	40	74.2	No	Ditto
-423	194,000	201,000	6	8.2	No	Cleavage
			Notched Specimens			
75		104,800		81.6	No	Shear
75		117,200		74.5	No	Shear and cleavage
75		103,200		77.7	No	Ditto
-323		198,200		4.7	Yes	Cleavage
-323		185,800		Nil	Yes	"
-323		195,400		Nil	Yes	"
-105		126,700		66.2	No	Shear and cleavage
-105		136,600		36.3	No	Cleavage
-105		124,700		49.7	No	"
-423		152,000		0	Yes	"

TABLE 27. TENSILE AND NOTCH TENSILE PROPERTIES OF RECRYSTALLIZED COLUMBIUM WITH 1000 PPM OXYGEN ADDED

Test Temperature, F	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent	Fracture at Maximum Load	Fracture Mechanism
			Unnotched Specimens			
75	61,000	64,600	44	83.7	No	Shear
75	58,500	67,200	40	79.3	No	"
-323	143,300	153,700	10	9.1	Yes	Cleavage
-323	158,900	168,000	(a)	3.3	Yes	"
-105	77,600	87,400	36	66.4	No	Shear and cleavage
-105	81,000	89,700	36	59.2	No	Cleavage
-423	(b)	156,000	(a)	6	Yes	"
			Notched Specimens			
75		96,200		85.0	No	Shear and cleavage
75		97,800		84.8	No	Ditto
75		104,000		84.6	No	"
-323		178,500		0	Yes	Cleavage
-323		161,800		0	Yes	"
-105		129,300		45.8	No	Shear and cleavage
-105		134,600		14.4	Yes	Ditto
-105		131,000		45.3	No	"
-423(c)		156,000		0	--	Cleavage

(a) Fracture outside 1/2-inch gage mark.
(b) Fracture before 0.2 per cent yield.
(c) Fracture in threads.

TABLE 28. TENSILE AND NOTCH TENSILE PROPERTIES OF WROUGHT, STRESS-RELIEVED COLUMBIUM WITH 150 PPM HYDROGEN ADDED

Test Temperature, F	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent	Fracture at Maximum Load	Fracture Mechanism
			Unnotched Specimens			
75	48,900	61,600	10	14.1	Yes	Cleavage
75	50,300	61,300	6	11.7	Yes	"
-323	131,300	142,100	4	4.8	Yes	"
-323	134,400	143,300	2	3.8	Yes	"
200	50,600	61,200	30	95.1	No	Shear
200	46,400	57,600	30	90.0	No	Shear and cleavage
300	48,600	56,700	28	94.1	No	Shear
-105	66,600	71,800	4(a)	3.5	Yes	Cleavage
-423	(b)	157,000	0	0	Yes	"
			Notched Specimens			
75		70,700		9.4	Yes	Cleavage
75		82,300		7.8	Yes	"
75		80,700		9.4	Yes	"
-323		183,300		0	Yes	"
-323		189,200		0	Yes	"
-323		169,700		0	Yes	"
200		87,400		93.8	No	Shear
200		84,600		93.8	No	"
200		88,000		94.2	No	"
300		84,800		97.1	No	"
300		82,600		97.1	No	"
-105		22,400		3.2	Yes	Cleavage
-423		149,000		0	Yes	"

(a) Fracture on 1/2-inch gage mark.

(b) Fracture before 0.2 per cent yield.

TABLE 29. TENSILE AND NOTCH TENSILE PROPERTIES OF RECRYSTALLIZED COLUMBIUM WITH 150 PPM HYDROGEN ADDED

Test Temperature, F	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent	Reduction, in Area, per cent	Fracture at Maximum Load	Fracture Mechanism
			Unnotched Specimens			
75	24,200	36,800	16	17.2	Yes	Cleavage
75	23,800	38,100	18(a)	15.7	Yes	"
-323	105,000	120,800	2(b)	0	Yes	"
-323	(c)	104,200	0(b)	0	Yes	"
200	21,800	34,800	42	94.1	No	Shear and cleavage
200	21,300	33,600	50	94.1	No	Ditto
300	16,400	28,900	44	95.1	No	"
-105	39,700	46,000	2	5.6	Yes	Cleavage
-423	(a)	137,000	0	0	Yes	"
			Notched Specimens			
75		38,800		4.8	Yes	Cleavage
75		37,900		9.4	No	"
75		38,800		6.4	Yes	"
-323		81,000		0	Yes	"
-323		90,400		0	Yes	"
-323		98,600		0	Yes	"
200		53,600		67.2	No	Shear
200		50,800		95.9	No	"
200		48,200		88.5	No	Shear and cleavage
300		42,700		94.2	No	Shear
300		45,200		93.8	No	"
-105		26,300		4.8	No	Cleavage
-423		78,600		0	Yes	"

(a) Fracture outside 1/2-inch gage mark.
(b) Fracture on 1/2-inch gage mark.
(c) Fracture before 0.2 per cent yield.

TABLE 30. TENSILE AND NOTCH TENSILE PROPERTIES OF WROUGHT, STRESS-RELIEVED COLUMBIUM WITH 300 PPM HYDROGEN ADDED

Test Temperature, F	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent	Fracture at Maximum Load	Fracture Mechanism
			Unnotched Specimens			
75	48,800	61,900	8'	8.7	Yes	Cleavage
-323	138,300	152,900	2	5.0	No	"
-323	137,200	151,300	6	11.8	No	"
-105	(a)	48,500	0(b)	2.6	Yes	"
-105	(.	57,700	0(b)	1.2	Yes	"
300	43,000	52,500	32	92.4	No	Shear and cleavage
200	45,000	55,800	36	88.4	No	Ditto
400	45,400	52,800	28	94.5	No	Shear
			Notched Specimens			
75		79,200		0	Yes	Cleavage
75		81,700		0	Yes	"
-323		120,500		0	Yes	"
-323		146,200		0	Yes	"
-323		111,800		0	Yes	"
-105		28,400		0	Yes	"
-105		34,400		0	Yes	"
-105		37,800		0	Yes	"
300		81,200		88.2	No	Shear
300		83,000		94.9	No	"
200		82,100		81.6	No	"
400		81,500		91.2	No	"

(a) Fracture before 0.2 per cent yield.
(b) Fracture on 1/2-inch gage mark.

TABLE 31. TENSILE AND NOTCH TENSILE PROPERTIES OF RECRYSTALLIZED COLUMBIUM WITH 300 PPM HYDROGEN ADDED

Test Temperature, F	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent	Fracture at Maximum Load	Fracture Mechanism
			Unnotched Specimens			
75	28,900	42,600	10[a]	8.7	Yes	Cleavage
-323	(b)	119,100	0	0.5	Yes	"
-105	(b)	43,100	0[a]	0.3	Yes	"
300	26,600	38,700	28[c]	96.8	No	Shear
200	27,300	39,500	46	88.4	No	"
400	24,400	43,100	10[c]	92.8	No	"
			Notched Specimens			
75		45,400		1.6	Yes	Cleavage
75		51,200		0	Yes	"
-323		93,100		0	Yes	"
-323	—	80,700		0	Yes	"
-105		57,700		0	No	"
-105		48,700		0	Yes	"
300		53,000		92.6	No	Shear
300		52,800		91.3	No	"
200		53,900		84.9	No	Shear and cleavage
400		46,600		91.3	No	Shear

(a) Fracture on 1/2-inch gage mark.
(b) Fracture before 0.2 per cent yield.
(c) Fracture outside 1/2-inch gage mark.

TABLE 32. TENSILE AND NOTCH TENSILE PROPERTIES OF WROUGHT, STRESS-RELIEVED TANTALUM-10W

Test Temperature, F	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent	Fracture at Maximum Load	Fracture Mechanism
			Unnotched Specimens			
75	127,100	136,000	30	83.8	No	Shear
75	124,700	133,500	32	82.6	No	"
-323	200,900	217,700	10 (a)	71.0	No	Shear and cleavage
-323	203,600	211,900	10 (a)	78.9	No	Shear
-105	154,800	163,400	34	86.2	No	"
-105	153,300	161,100	32	80.2	No	"
-423	227,000	236,000	34	72	No	Shear and cleavage
			Notched Specimens			
75		194,000		87.0	No	Shear
75		190,900		83.6	No	"
75		193,700		85.0	No	"
-323		288,500		75.4	No	"
-323		290,100		74.5	No	"
-323		290,100		72.1	No	"
-105		222,100		88.9	No	"
-105		221,700		88.2	No	"
-105		225,200		88.0	No	"
-423		357,000		56.5	No	Shear and cleavage

(a) Fracture outside 1/2-inch gage mark.

TABLE 33. TENSILE AND NOTCH TENSILE PROPERTIES OF RECRYSTALLIZED TANTALUM-10W

Test Temperature, F	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent	Fracture at Maximum Load	Fracture Mechanism
			Unnotched Specimens			
75	77,600	90,700	34	50.6	No	Shear and cleavage
75	75,300	89,400	36	66.3	No	Shear
-323	142,700	163,800	18[a]	71.7	No	"
-323	141,000	162,400	32	71.8	No	Shear and cleavage
-105	103,400	108,400	14	18.5	No	Ditto
-105	104,700	112,100	28	39.7	No	"
-105	102,500	114,100	36	88.5	No	"
75	75,300	88,500	46	90.7	No	"
-423	164,000	179,000	18	64	No	"
			Notched Specimens			
75		133,300		89.6	No	Shear
75		137,600		84.0	No	"
75		138,600		87.8	No	"
-323		230,100		67.1	No	Shear and cleavage
-323		226,600		55.7	No	Ditto
-323		229,000		54.7	No	"
-105		170,900		87.6	No	Shear
-105		167,200		90.1	No	"
-105		168,900		89.6	No	"
-423		290,000		18.3	Yes	Cleavage

(a) Fracture outside 1/2-inch gage mark.

TABLE 34. TENSILE AND NOTCH TENSILE PROPERTIES OF WROUGHT, STRESS-RELIEVED F-48, Cb ALLOY

Test Temperature, F	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent	Fracture at Maximum Load	Fracture Mechanism
			Unnotched Specimens			
75	104,500	130,700	20	32.4	No	Shear and cleavage
-105	142,700	155,700	4	6.6	Yes	Ditto
-323	184,100	191,900	(a)	5.0	Yes	Cleavage
200	104,500	128,700	22	50.3	No	Shear
300	88,900	108,800	28	59.2	No	Shear and cleavage
0	113,200	136,300	26	42.8	No	Cleavage
-423	214,000	219,100	(b)	0	Yes	"
			Notched Specimens			
75		188,700		0	Yes	Shear and cleavage
75		149,000		0	Yes	Cleavage
-105		160,300		0	Yes	"
-105		162,400		0	Yes	"
-323		115,900		1.6	Yes	"
-323		113,600		3.2	Yes	"
200		176,000		11.0	Yes	Shear and cleavage
200		158,800		4.8	Yes	Ditto
300		165,600		25.6	No	Cleavage
300		130,800		1.7	Yes	"
300		172,400		9.4	Yes	"
0		177,000		3.2	Yes	"
0		117,200		0	Yes	"
-423		128,000		1.7	Yes	"

(a) Multiple fracture, piece missing.
(b) Multiple fracture, one break on gage mark, one break in.

TABLE 35. TENSILE AND NOTCH TENSILE PROPERTIES OF RECRYSTALLIZED F-48, Cb ALLOY

Test Temperature, F	0.2 Per Cent Offset Yield Strength, psi	Ultimate Tensile Strength, psi	Elongation, per cent	Reduction in Area, per cent	Fracture at Maximum Load	Fracture Mechanism
			Unnotched Specimens			
75	108,800	114,500	6[(a)]	3.1	Yes	Cleavage
-105	(b)	133,000	0	0	Yes	"
200	79,400	103,800	26	44.7	No	"
-323	(b)	179,000	0	1.2	Yes	"
-323	183,300	184,300	(c)	0.2	Yes	"
300	75,600	97,600	30	52.0	No	"
			Notched Specimens			
75		125,500		1.6	Yes	Cleavage
75		140,200		0	Yes	"
-105		101,800		0	Yes	"
-105		119,000		0	Yes	"
200		115,900		0	Yes	"
200		119,700		0	Yes	"
-323		112,100		0	Yes	"
-323		113,400		0	Yes	"
300		136,000		4.7	Yes	"
300		125,900		7.8	Yes	"

(a) Multiple fracture, one break on gage mark, one break in.
(b) Broke before 0.2 per cent yield.
(c) Multiple fracture, piece missing.

Aeronautical Systems Division, Wright-Patterson Air Force Base, Ohio. Rpt. No. ASD-TR-61-474. NOTCH SENSISTIVITY OF REFRACTORY METALS. Final Rpt, January 1962, 128p incl. illus., tables.

Unclassified Report

The effects of interstitial oxygen and hydrogen on the tensile and notch tensile properties of tantalum and columbium were investigated. The tensile and notch tensile properties of Ta-10W and F-48 columbium alloy were determined also. Oxygen and hydrogen additions resulted in notch-sensitive behavior at higher temperatures than for pure Ta and Cb, and, similarly, transit-

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ion temperatures are increased by the interstitial additions. The F-48 alloy also shows notch-sensitive behavior at higher temperatures and higher transition temperatures than columbium. The Ta-10W alloy was not notch sensitive, and retained excellent ductility at -420 F.

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